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EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION OF COATED SUPERALLOYS IN MARINE ENVIRONMENT

Progress Report No. 3
October 1, 1966 to December 31, 1966

By
H. T. Quigg and R. M. Schirmer

Prepared under Contract NOw 66-0263-d for the
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by Phillips Petroleum Company
Bartlesville, Oklahoma

April 1967

Research Division Report 4706-67R

PHILLIPS PETROLEUM COMPANY

RESEARCH DIVISION

BARTLESVILLE, OKLAHOMA

PROGRESS REPORT NO. 3
FOR
NAVAL AIR SYSTEMS COMMAND CONTRACT NOw 66-0263-d

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S U M M A R Y

An experimental investigation is in progress to determine whether the 0.4 per cent by weight of sulfur allowed in JP-5 fuel is a safe level for the protection of turbine-blade alloys used in high-performance engines.

Specimens of a nickel-base alloy (Inconel 713C) and the alloy with either an aluminum-diffusion coating (Misco MDC-1) or an aluminum-chromium-diffusion coating (Misco MDC-9) were exposed to vitiated air from the Phillips 2-inch combustor (60 air-fuel ratio) at high pressure (15 atmospheres), high velocity (745 ft/sec), and high temperature (2000 F) with 5-hour sampling periods and test durations of up to 55-hours. Tests were conducted at all combinations of three levels of sulfur in fuel ($<$0.0040, 0.040 and 0.40 weight per cent) with two levels of "sea salt" in air (zero and 1.0 ppm). With one coating (Misco MDC-1), tests were also conducted with 10.0 ppm "sea salt" in air and three levels of sulfur in fuel ($<$0.0040, 0.40 and 4.0 weight per cent). The use of specimen weight-loss as a measure of hot-corrosion attack was validated by metallographic examination which showed the absence of deep intercrystalline attack.

Exponential equations of weight-loss with time have been developed and statistically-significant effects have been identified at a 95 per cent confidence level. In all comparisons the removal of "sea salt" from air significantly decreased the relative rate of corrosion; thus, indicating sea salt to be a primary-causative agent of hot corrosion. The effect of sulfur in fuel varied with the superalloy coating and the absence or presence of "sea salt". In the absence of "sea salt" in air a reduction of sulfur in fuel from the present limit to either 0.040 or $<$0.0040 weight per cent decreased attack with one coated superalloy and increased attack with the other. In the presence of 1.0 ppm "sea salt" in air, a reduction in sulfur to 0.040 weight per cent had no significant effect on attack; however, a reduction to $<$0.0040 weight per cent significantly decreased the relative rate of attack for both coated superalloys.

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BARTLESVILLE, OKLAHOMA

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EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION OF COATED SUPERALLOYS IN MARINE ENVIRONMENT

1. INTRODUCTION

The corrosion of hot-section parts in modern aircraft-turbine engines is one of the factors that determines the time before overhaul. With operation in a marine environment it becomes a major factor in limiting engine life. Various terms have been used to identify this accelerated attack on the superalloys from which hot-section parts are fabricated. We favor the term "hot corrosion," and will use it in this report to indicate the attack by sea salt on superalloys at high temperature.

Considerable metal loss can be sustained by hot-section parts before failure because hot corrosion advances on a broad front. The attack is led by penetration of randomly dispersed light-grey globules of metalic sulfide. The formation of these sulfides is associated with changes, characterized by chromium depletion, in the surface composition of the alloy. Rapid oxidation of the weakened layer of the alloy follows. Because of the prominent band of precipitated sulfides preceding surface oxidation, hot corrosion is frequently identified as "sulfidation." This has focused attention on the sulfur content of the fuel as being the principal causative agent of hot corrosion. If so, hot corrosion could be controlled by fuel specification. More restrictive limitations on the amount of sulfur allowed in aviation-turbine fuels have been proposed for this purpose, particularly for naval operations.

Most specifications for aviation-turbine fuel allow a sulfur content of 0.40 weight per cent. A significant reduction in the sulfur limit would certainly decrease the amount of available fuel. Also, a more restrictive specification carries with it the potential of higher cost, and a modest increase can amount to a substantial sum because of the large volume involved. Therefore, proposals to lower the sulfur content of aviation-turbine fuel must be approached with caution.

Many investigators feel that deposition of sodium sulfate on the metal surface is a normal precursor to hot corrosion. Some have reasoned that sodium from sea salt and sulfur from fuel combine to form the objectionable sodium sulfate. However, compounds other than sodium chloride are present in sea salt. Sea salt contains 11 per cent by weight of sodium sulfate. Control of hot corrosion by reducing sulfur in fuel to remove one of the critical ingredients from the salt-sulfur combination ignores the sulfur in sea salt.

For clarification, the Naval Air System Command has supported our work to determine whether the maximum sulfur content of 0.40 weight per cent, currently allowed in grade JP-5 aviation-turbine fuel, is a safe level for protection of turbine alloys in high performance engines when operated in a marine environment.

In previous studies, reported in detail by Schirmer and Quigg (1), we attempted to simulate the environment in the turbine section of an aircraft engine with respect to temperature, velocity, pressure, and stoichiometry by use of a high-pressure test facility, originally developed for evaluating the combustion characteristics of aviation-turbine fuels. The test programs included the effect of three levels of sulfur in fuel on hot corrosion of six superalloys, one of which was aluminum coated, at five levels of temperature and three levels of "sea salt" in air. The five hours of test duration used was sufficient for extensive corrosion of all of the uncoated superalloys under some conditions. It was concluded that an order-of-magnitude reduction (0.40 to 0.040 weight per cent) in sulfur content would not decrease hot corrosion, and no change in fuel specification was indicated. The aluminum-diffusion coating (Misco MDC-1) on Inconel 713C resulted in a material immune to attack under the conditions of exposure used during this investigation; therefore, no measure of the effect of sulfur in fuel on the hot corrosion of a coated superalloy was obtained.

Coating of superalloys is one method which has been demonstrated to reduce attack by sea salt. To measure the effect of sulfur in fuel on hot corrosion and the extent of protection afforded by a coating, an exploratory "life" test (2) was undertaken with Misco MDC-1 coated Inconel 713C. The test duration was extended to obtain extensive corrosion at the 2000 F test condition with 10.0 ppm "sea salt" in air. Two tests were included in the investigation; one test with the specification-maximum concentration of 0.40 weight per cent sulfur in fuel, and the other with an essentially sulfur-free fuel ($<$0.0040 weight per cent). It was found that deterioration of the coating had progressed sufficiently in 10 to 20 hours of exposure to evidence catastrophic rates of corrosion. With the rapid rate of attack, the data were rather badly scattered; but, there was evidence that sulfur in the fuel inhibited hot corrosion at this relatively severe condition.

The purpose of our current investigation is to examine in greater detail the effect of sulfur in fuel on the hot corrosion of coated superalloys in a marine environment. Three levels of sulfur in fuel (<0.0040, 0.040, and 0.40 weight per cent) and two levels of "sea salt" in air (zero and 1.0 ppm) are included in the program, with exposure at the 2000 F test condition used in previous studies. In addition, tests were conducted with 10.0 ppm "sea salt" in air, at three levels of sulfur in fuel (<0.0040, 0.40, and 4.0 weight per cent), with one of the coated superalloys. Test duration has been extended up to 55-hours, with removal of test specimens at 5-hour intervals to obtain a series of specimens for each combination of sulfur in fuel and "sea salt" in air.

A concentration of 1.0 ppm "sea salt" in air, which is realistic for many naval-aircraft operations, was selected for the major portion of the program to simulate a marine environment. It was hoped that a decrease in experimental error would accompany the reduction in severity of attack from the catastrophic levels experienced with a concentration of 10 ppm during the exploratory program.

Work has been completed showing the effect of sulfur in fuel on the hot corrosion of one superalloy (Inconel 713C) and two different coating-alloy systems (Misco MDC-1 and Misco MDC-9 on Inconel 713C). Inconel 713C is a nickel-base alloy. Misco MDC-1 is an aluminum-diffusion coating containing non-metallic dispersions, and Misco MDC-9 is a composite coating rich in aluminum and chromium. An analysis of these data is presented in this report.

2. CONCLUSIONS

Experiments have been conducted, using a high-pressure burner rig, to evaluate the effect of sulfur in JP fuel on the hot corrosion of coated turbine-blade materials. The durability of two diffusion-type coatings, differing in composition, have been investigated at a 2000 F test condition, which simulates exposure in a high-performance engine when operated in a marine environment. Also, for reference purposes, data have been obtained in the absence of "sea salt", and with the uncoated superalloy.

The use of weight loss by test specimens as a measure of hot-corrosion attack was validated by metallographic examination, which showed corrosion advancing on a broad front without deep intercrystalline penetration. Electro-cleaning in a hot-caustic bath was used to remove accumulated metal-oxidation products and sea-salt residues from Misco MDC-1 coated Inconel 713C specimens, and also from the uncoated superalloy; however, it was found to be too severe for the Misco MDC-9 coated Inconel 713C specimens, and they were sonically-cleaned in a water bath.

Exponential equations of weight loss with time were developed which permit evaluating the effects of three levels of sulfur in fuel ($<$ 0.0040, 0.040, and 0.40 weight per cent) and two levels of "sea salt" in air (zero and 1.0 ppm). For one of the coating-alloy systems the effect of "sea salt" was extended to a third level (10 ppm). The following conclusions can be drawn at a 95 per cent confidence level.

A. <u>In all comparisons</u>, a reduction in the concentration of "sea salt" in air, or its removal from the air, significantly <u>decreased</u> the relative rate of corrosion; thus, indicating sea salt to be a primary-causative agent in the hot corrosion of both coated and bare superalloys in a marine environment.

B. <u>In the absence of "sea salt" in air</u>, a reduction of sulfur in fuel from 0.40 to either 0.040 or $<$0.0040 weight per cent:

(a) significantly <u>decreased</u> the relative rate of corrosion on Misco MDC-1 coated Inconel 713C,
(b) had no significant effect on the relative rate of corrosion for Misco MDC-9 coated Inconel 713C, but significantly <u>increased</u> the level of attack on this coated superalloy, and
(c) had no significant effect on the relative rate of corrosion for uncoated Inconel 713C.

While statistically significant differences in the relative rates of corrosion were detected, the levels of attack were very low and may not be of practical significance.

C. In the presence of 1.0 ppm "sea salt" in air, a reduction of sulfur in fuel from 0.40 to 0.040 weight per cent:

(a) had no significant effect on the relative rate of corrosion for either Misco MDC-1 or MDC-9 coated Inconel 713C, and
(b) had no significant effect on the relative rate of corrosion for uncoated Inconel 713C, but significantly decreased the level of attack on this uncoated superalloy.

However, a further reduction of sulfur in fuel from 0.40 to <0.0040 weight per cent significantly decreased the relative rate of corrosion on both Misco MDC-1 and MDC-9 coated Inconel 713C, as well as on the uncoated superalloy.

3. RECOMMENDATIONS

The primary objective of this investigation is to determine whether the 0.4 weight per cent sulfur currently allowed in grade JP-5 fuel is a safe level for the protection of turbine blades in aircraft engines of advanced design.

Programs have been completed which allowed an evaluation of the effect of sulfur concentration in fuel on the durability of bare superalloys (1). It was concluded that a reduction in sulfur content by an order of magnitude, to 0.04 weight per cent, would not reduce hot corrosion significantly. Therefore, it was recommended that no change in the sulfur limit for JP-5 be made to alleviate hot-corrosion attack on turbine blades.

That recommendation was tempered by another to extend this investigation to evaluate the effect of fuel sulfur on the protection afforded by coatings. Those data were sought because superalloys having the most desirable physical properties are more susceptible to hot corrosion and require coatings for satisfactory durability.

Programs have been completed which allow an evaluation of the effect of sulfur concentration in fuel on the durability of coated superalloys. Our analysis of the data from these programs shows one case where a reduction of sulfur in fuel from the present limit to 0.04 weight per cent significantly decreased hot corrosion, and another case where the attack was significantly increased. This indicates that our previous recommendation based upon a study of bare superalloys, can be extended to coated superalloys.

It is emphasized that additional data are required with other coating-superalloy systems and other exposure temperatures before making a final recommendation.

4. RESULTS AND DISCUSSION

This experimental investigation was designed to permit comparisons showing the effect of sulfur concentration in JP-5 fuel on the hot corrosion of coated superalloys in a marine environment. To characterize the principal levels of interest, data were obtained with three concentrations of sulfur in the fuel (0.40, 0.040, and $<$ 0.0040 weight per cent), representing specification maximum, production median, and essentially sulfur-free fuels. A marine environment was simulated by the addition of "sea salt" to the air (1.0 ppm). Two diffusion-type coatings were used in this investigation; one rich in aluminum (Misco MDC-1) and the other rich in aluminum and chromium (Misco MDC-9), both applied to the same nickel-base alloy (Inconel 713C). Also, for reference purposes, data were obtained in the absence of "sea salt", and with the uncoated superalloy.

In previous studies (1) aluminum-coated specimens (Misco MDC-1 on Inconel 713C) were immune to attack under all combinations of temperature, sulfur in fuel, and "sea salt" in air with the standard 5-hour test duration. The present experiments were designed to stress the coating to failure by extending test duration and, thereby, permit evaluating the effect of sulfur in fuel on the durability of coated superalloys. Using a 1-hour cyclic procedure, test specimens were removed, with replacement, at 5-hour intervals to obtain a series of specimens with exposures of up to 55 hours duration.

To simulate the environment in the turbine section of an aircraft engine, a cascade holder supporting six test specimens was mounted in the exhaust section of Phillips 2-inch combustor. The facility was operated to obtain exposure of the test specimens at high pressure (15 atmospheres), high temperature (2000 F), high velocity (745 feet per second), and realistic stochiometry (60 air-fuel ratio).

Further details concerning the test equipment, test materials, and test programs are presented in Appendices 2, 3, and 4, respectively, which are Sections 9, 10, and 11 of this report.

There is no generally-accepted criterion at the present time for determining the effective life of a coating on a superalloy. Evaluations have been based on weight loss by specimens, visual appearance of specimens, and combinations of both.

In a previous investigation (3) an equation was developed for calculating the depth of penetration of attack from weight loss, assuming a uniform depth of penetration on all surfaces. From this equation the weight loss for a uniform loss of 0.0002 inches (the thickness of the coating) from all surfaces of the specimen was calculated to be 790 mg, or 39 mg/cm^2. While hot corrosion characteristically advances on a broad front, the attack is usually somewhat localized on the surface; thus, the coating would probably be penetrated before all of it is removed, and a value of 39 mg/cm^2 would be high.

Whitfield and Parzuchowski (4) established a weight loss of 0.100 grams as the criteria for failure of coated specimens. Using dimensions of the corrosion bars given by Danek (5), the surface area of their specimens is calculated to be 20.00 cm^2. From these figures we calculate the criteria for failure of the coating to be 5.0 mg/cm^2.

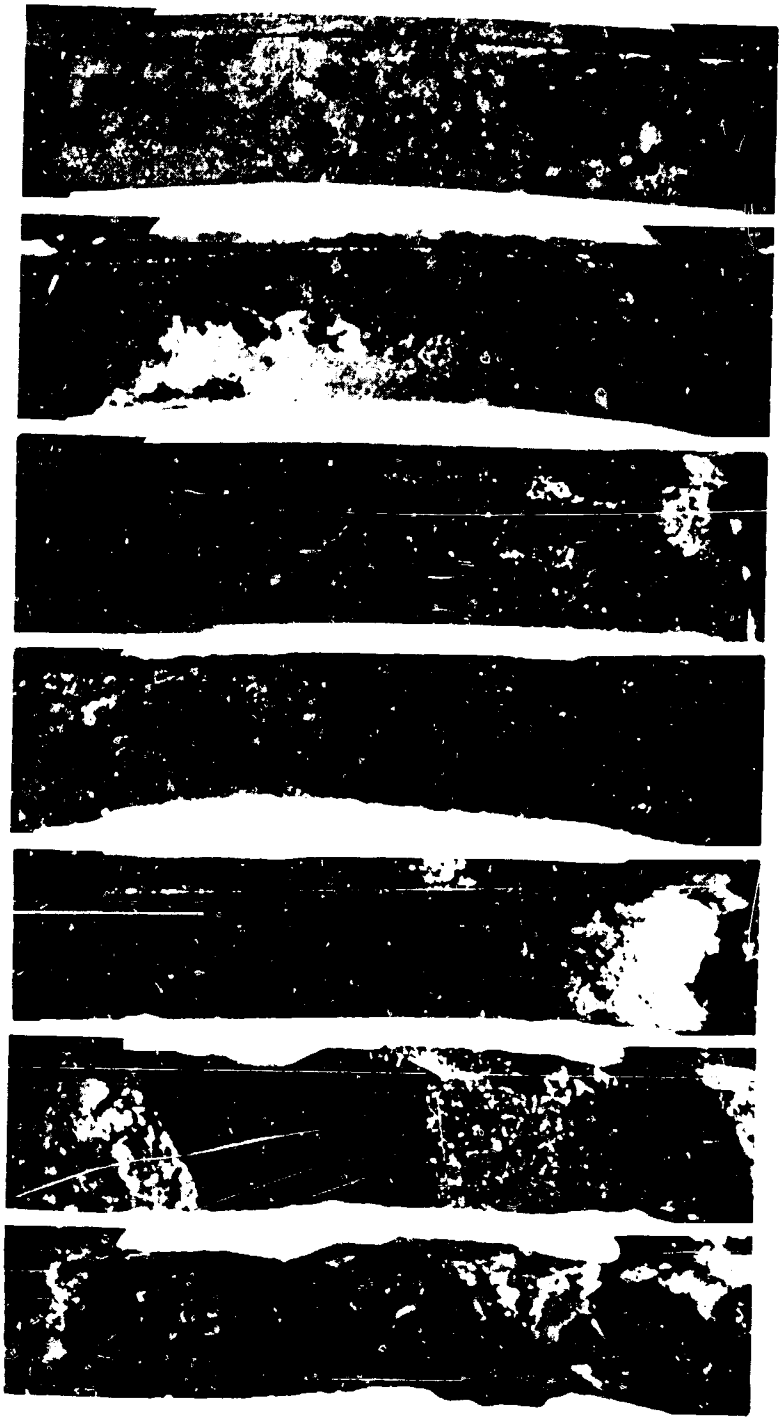
Visual examination of test specimens from our programs indicate that coatings have been penetrated on specimens showing "Light Surface Attack: or "Light Edge Attack", based on a visual-rating scale which will be described later. A weight loss of 5 mg/cm^2 falls in the range of weight losses obtained for visual ratings of "Light" and would represent a reasonable criteria for coating failure.

In this report test specimens will be examined with respect to both weight loss and visual attack to evaluate the effect of sulfur in fuel and "sea salt" in air on hot corrosion.

4.1. Visual Ratings

The condition of Misco MDC-1 coated Inconel 713C specimens following extended exposure at the 2000 F test condition, both with and without "sea salt" in air, is shown in Figure 1. These photographs were taken with the specimens still mounted in the cascade holder to show the undisturbed accumulation of deposits. Typically, the specimens are quite clean in the absence of "sea salt"; however, with ingestion of "sea salt", the attack is accelerated and considerable accumulation of deposits was observed. These deposits tend to flake-off the test specimens during cooling to room temperature, which indicates a significant difference in their coefficients of expansion. Analysis of deposits accumulated at the 2000 F condition in the presence of 10.0 ppm "sea salt" in air are presented in Table 1, and show them to be metal oxide corrosion products with little evidence of "sea salt" residue or reaction products. Following removal from the cascade holder, specimens were cleaned in preparation for measurement of the extent of attack.

Visual ratings of each Misco MDC-1 coated Inconel 713C test specimen are shown in Tables 10 to 12 of Appendix 1, and for the Misco MDC-9 coated Inconel 713C specimens in Tables 13 and 14 of Appendix 1. The system used in obtaining these ratings was developed from an examination of the coated specimens from these programs, and is shown in Table 2. The photographic standards used for rating surface and edge attack of the specimens are shown in Figures 2 and 3.



EXPOSURE TIME, HR	FUEL SULFUR, %	SEA SALT IN AIR, PPM
55	< 0.0040	NONE
55	0.40	
35	< 0.0040	1.0
40	0.40	
5		
10	< 0.0040	10.0
15		

2X MAGNIFICATION OF MISCO MDC-1 COATED INCONEL 713C SPECIMENS STILL MOUNTED IN HOLDER AFTER EXPOSURE IN PHILLIPS RIG

FIGURE 1
ACCUMULATION OF DEPOSITS ON SPECIMENS AT 2000 F TEST CONDITION

TABLE 1

ANALYSIS OF SCALE COMPOSITION [a]

General Composition [b]	Found, wt % [c]
NiO(Bunsenite)	Major Component
$Ni(AlCr)_2O_4$ [d]	Minor Component
Na_2SO_4	ND
Element [e]	
S	D
K	D
Cl	ND

Element [f]	Limit of Detection, wt %		Element [f]	Limit of Detection, wt %	Found, wt % [c]
Ni	0.1	> 10	Sn	0.1	0.01-0.1
Cr	0.01	> 1	Cu	0.01	0.01-0.1
Al	0.01	> 1	Zr	0.01	0.01-0.1
Mo	0.1	> 1	Sr	0.1	0.01-0.1
Mg	0.001	> 1	Ba	0.01	ND
Na	0.1	> 1	B	0.001	ND
Ca	0.01	0.3-3.0	P	1.00	ND
Co	0.1	0.1-1.0	Pb	0.01	ND
Ti	0.01	0.1-1.0	Cd	0.1	ND
Fe	0.01	0.1-1.0	Zn	0.1	ND
Mn	0.001	0.01-0.1	V	0.1	ND
Si	0.01	0.01-0.1	Li	0.1	ND

Notes:

(a) Deposits scraped from surface of four test specimens of Misco MDC-1 coated Inconel 713C alloy after exposure for 15 hours at 2000 F test condition with 10 ppm "sea salt" in air and 0.4 wt % sulfur in fuel. Deposit flaked-off during cooling, but was evident in some areas to a depth of about 1/16 inch.

(b) X Ray Diffraction Analysis.

(c) Concentrations will be placed in proper range at least 90% of time. ND means the concentration is less than the limit of detection. D means detected but concentration was not determined. > means greater than.

(d) Estimated from similarity of pattern to $Fe(AlCr)_2O_4$.

(e) X-Ray Flourescence Analysis. (f) Emission Spectrograph Analysis.

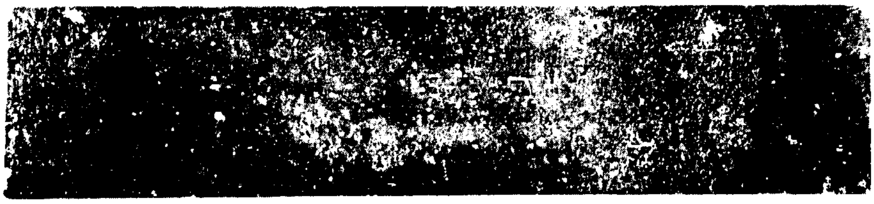
TABLE 2

SYSTEM FOR VISUAL RATING OF ATTACK ON COATED TEST SPECIMENS

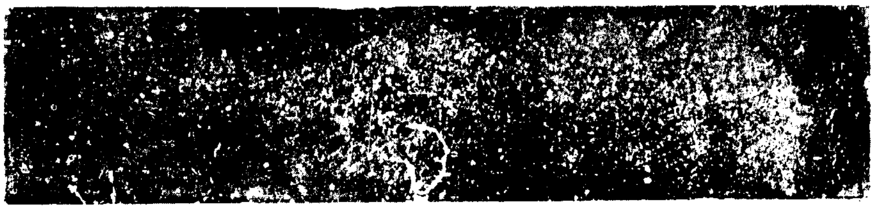
Rating Code	Rating Value	Severity of Attack
N	10	None
VL	8	Very Light
L	6	Light
M	4	Medium
H	2	Heavy
		Nature of Attack
S		Surface
E		Edge
		Condition of Specimen
C		Surface Cracks

Example:

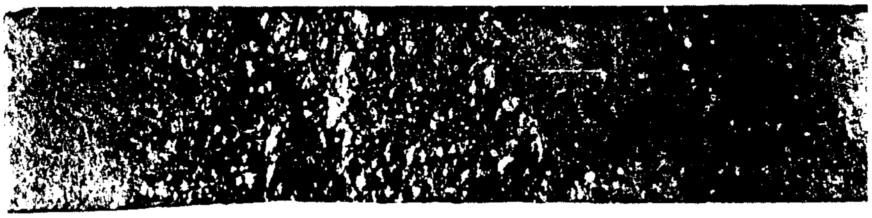
VLS = Very Light Surface Attack



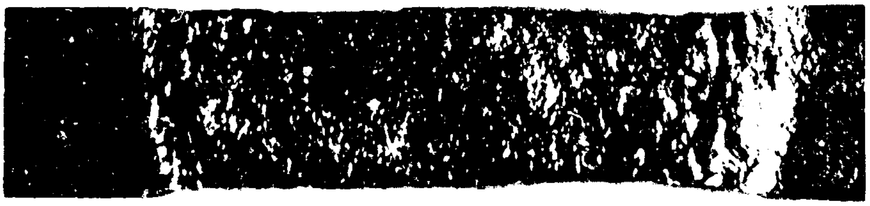
VERY LIGHT



LIGHT

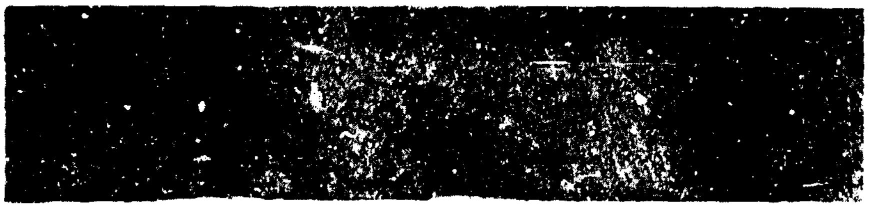


MEDIUM



HEAVY

FIGURE 2
STANDARDS FOR VISUAL RATING OF SURFACE ATTACK
ON COATED SPECIMENS (2X)



VERY LIGHT

LIGHT

MEDIUM

HEAVY

FIGURE 3
STANDARDS FOR VISUAL RATING OF EDGE ATTACK
ON COATED SPECIMENS (2X)

Arbitrary merit ratings were assigned to the visual ratings which are shown in Table 2. Using these, plots of logarithms of visual ratings vs logarithms of the weight losses per unit area were made for Misco MDC-1 coated Inconel 713C and Misco MDC-9 coated Inconel 713C specimens, and are shown in Figures 4 and 5. As expected, visual ratings decrease as weight loss increases.

To further illustrate the effect of the exposure variables in this investigation on appearance, photomacrographs (2X) of representative Misco MDC-1 coated Inconel 713C test specimens are shown in Figures 6 and 7; Misco MDC-9 coated Inconel 713C in Figure 8, and uncoated Inconel 713C in Figure 9. By comparison of the extent of attack evident for similar exposures in Figures 6, 8, and 9, it is obvious that the coatings protect the superalloy from hot corrosion. Increased attack on both coated and bare specimens is evident in the presence of 1.0 ppm "sea salt", over that in the absence of "sea salt". In the presence of 1.0 ppm "sea salt" a reduction in the concentration of sulfur in fuel decreased attack; however, in the presence of 10.0 ppm "sea salt" a reduction in the concentration of sulfur in fuel increased attack.

The visual rating is a subjective value, while weight loss is a physical measurement that should be associated with a smaller error. Also, the visual rating groups the data at only five levels, with the proposed system shown in Table 2, while the use of weight loss does not require the grouping of data. For these reasons, and in the light of metallographic examinations which established the mode of attack and will be discussed later, it was decided that weight loss measurements would be used in evaluating the effects of sulfur in fuel and "sea salt" in air on hot corrosion.

During the visual rating of the Misco MDC-9 coated Inconel 713C specimens it was noted that they had a tendency to develop visible surface cracks as the result of exposure to hot gases. Similar cracks were not observed with the Misco MDC-1 coated Inconel 713C specimens, nor in an exploratory program with Misco MDC-9 coated Inconel 713C specimens. The cracks usually developed on the leading edge of the specimens, and became more evident with increasing exposure time. This is illustrated in Figure 10 by the set of specimens accumulated from the test with the medium-sulfur fuel. The accelerated rate of corrosion which accompanied the addition of "sea salt" to this environment appeared to have little affect on the occurance and development of cracks, as illustrated in Figure 11 by the set of specimens accumulated at otherwise comparable conditions of exposure.

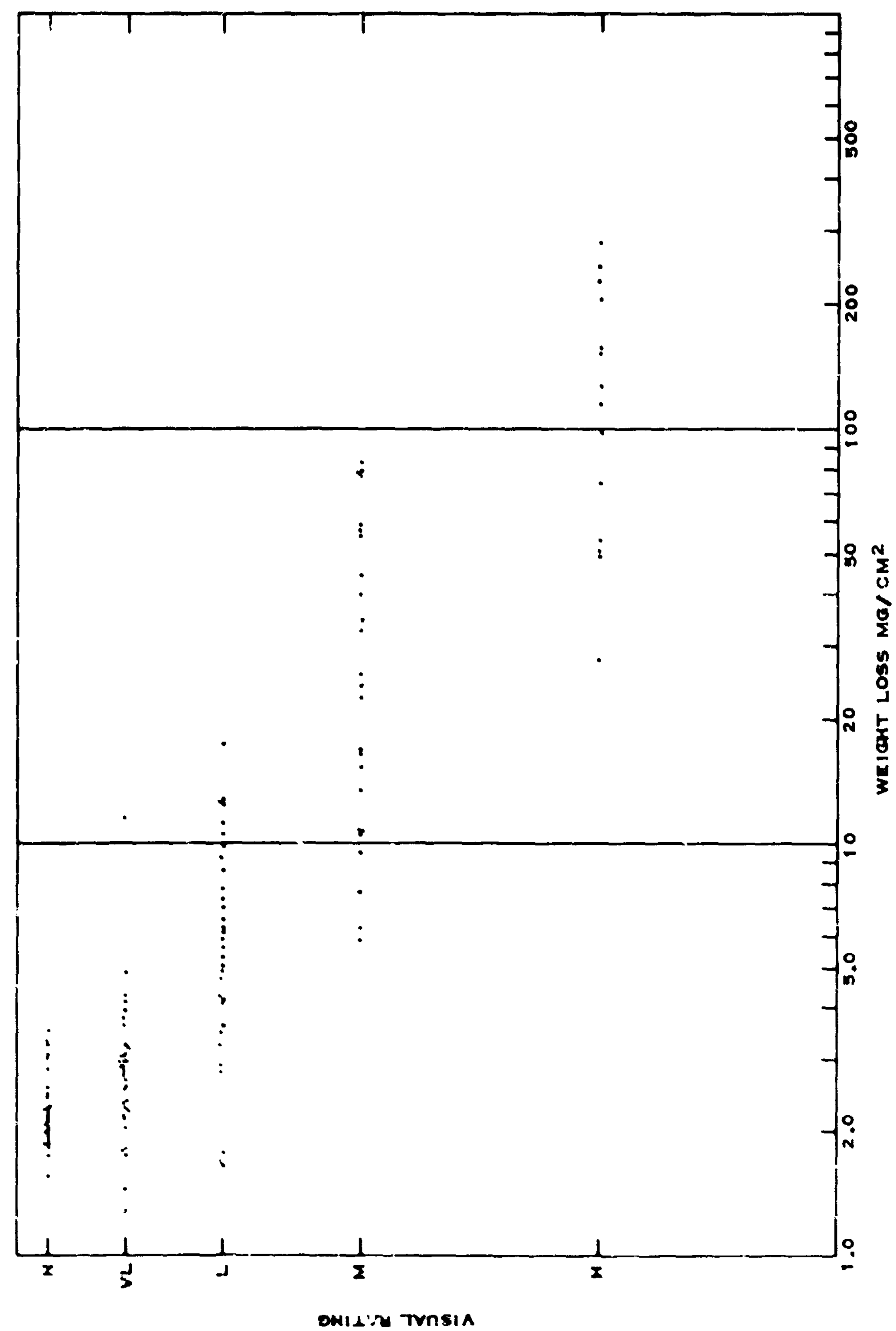


FIGURE 4
RELATIONSHIP BETWEEN VISUAL RATING AND WEIGHT LOSS
FOR MISCO MDC-1 COATED INCONEL 713C

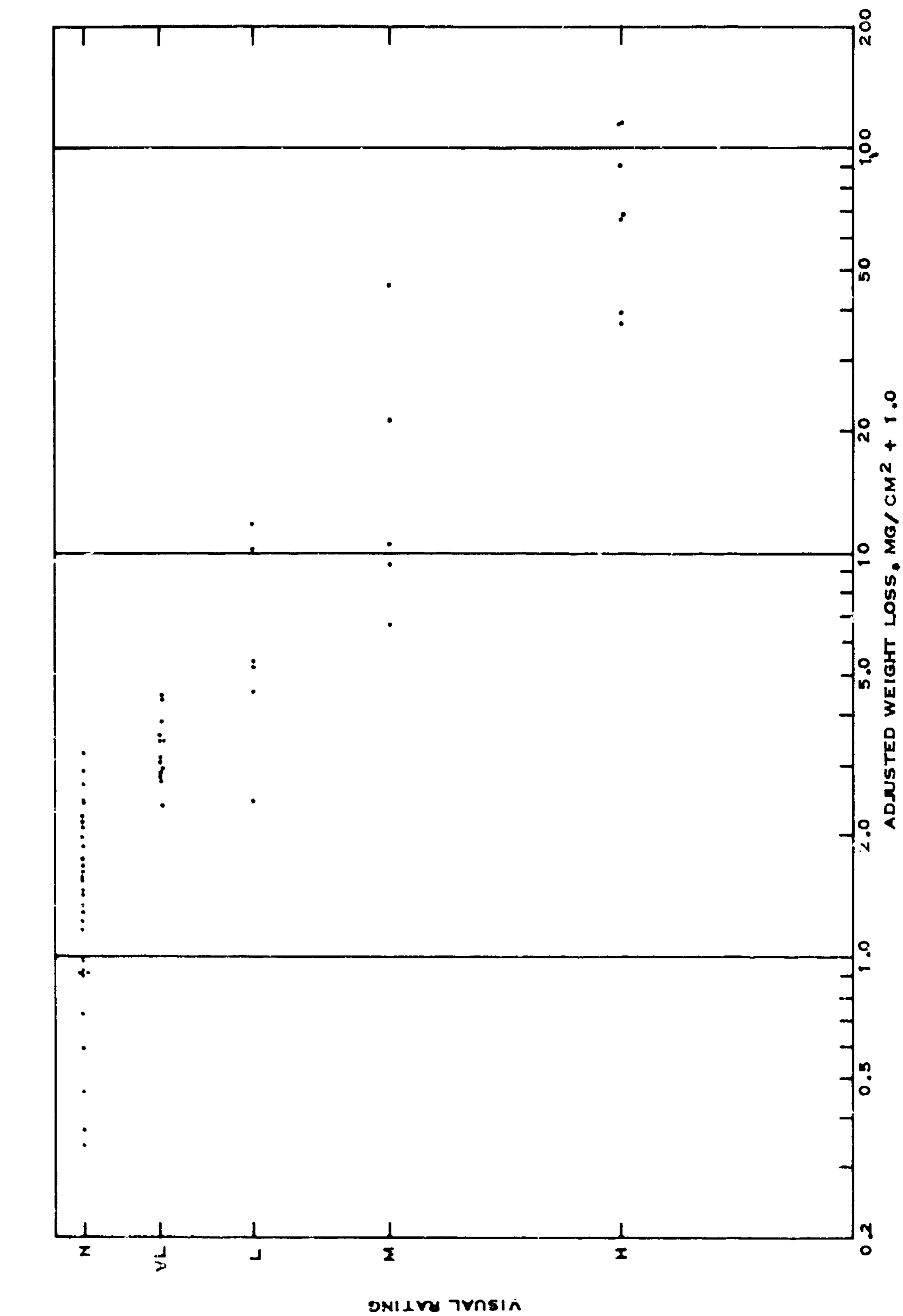
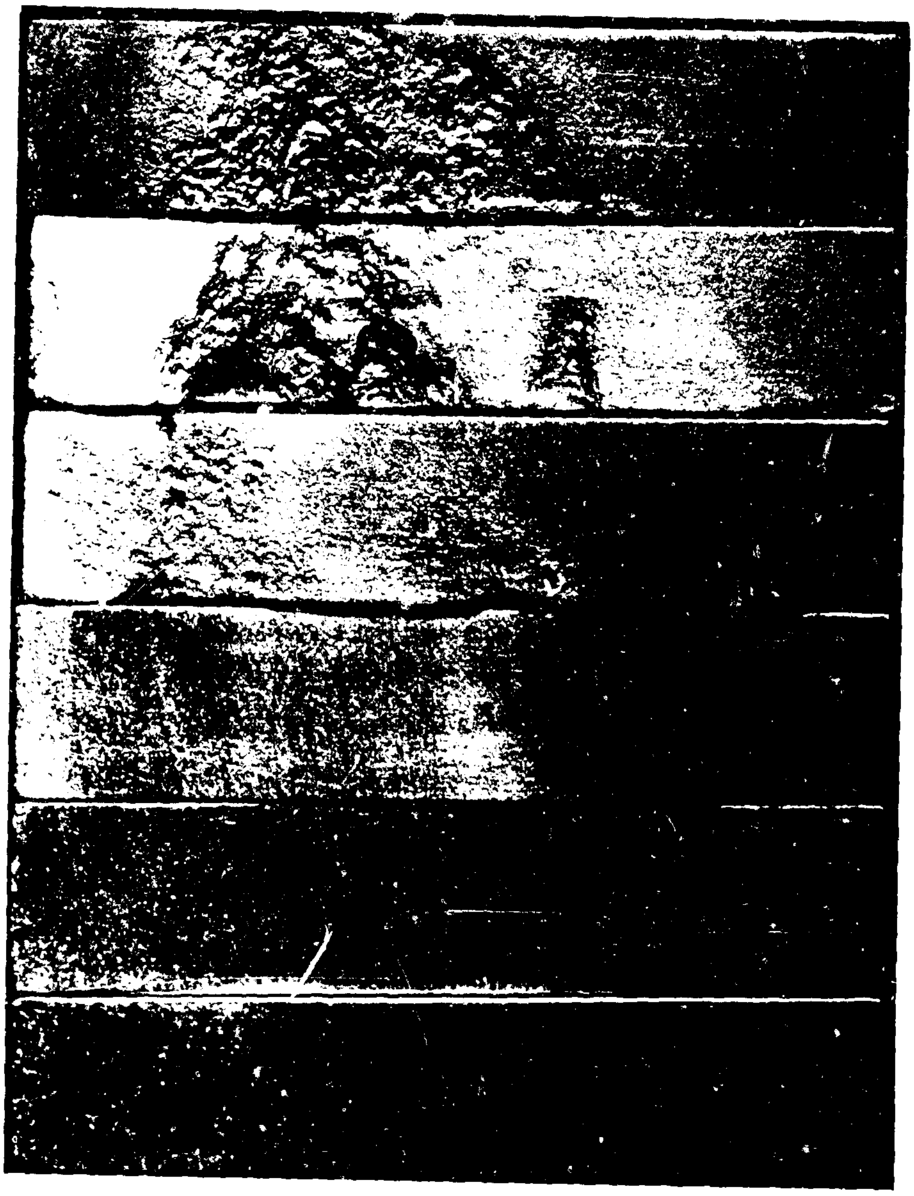


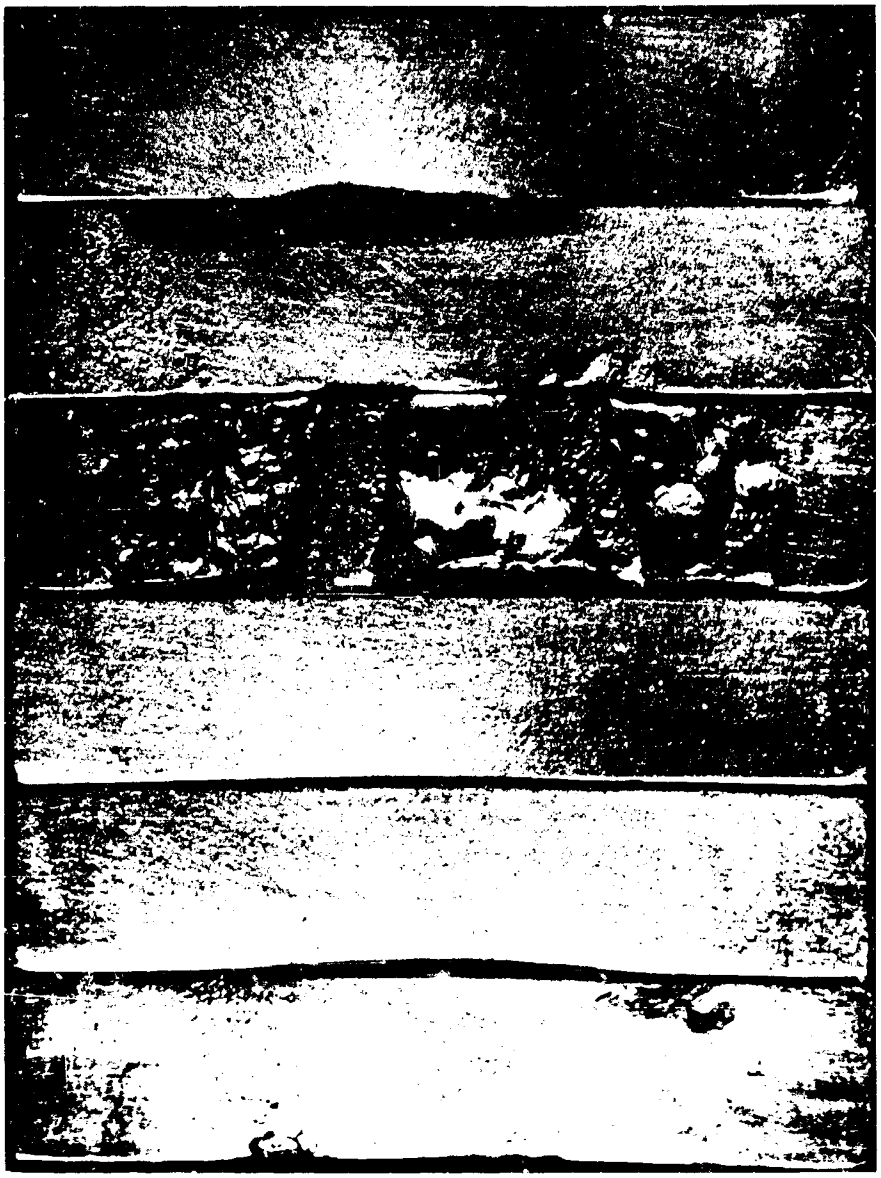
FIGURE 5
RELATIONSHIP BETWEEN VISUAL RATING AND WEIGHT LOSS FOR MISCC MDC-9 COATED INCONEL 713C



SULFUR IN FUEL, WT %	SEA SALT IN AIR, PPM
0.40	1.0
0.040	1.0
<0.0040	1.0
0.40	NONE
0.040	NONE
<0.0040	NONE

2X MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 6
MISCO MDC 1 COATED INCONEL 713C SPECIMENS
AFTER 25 HOURS AT 2000 F TEST CONDITION

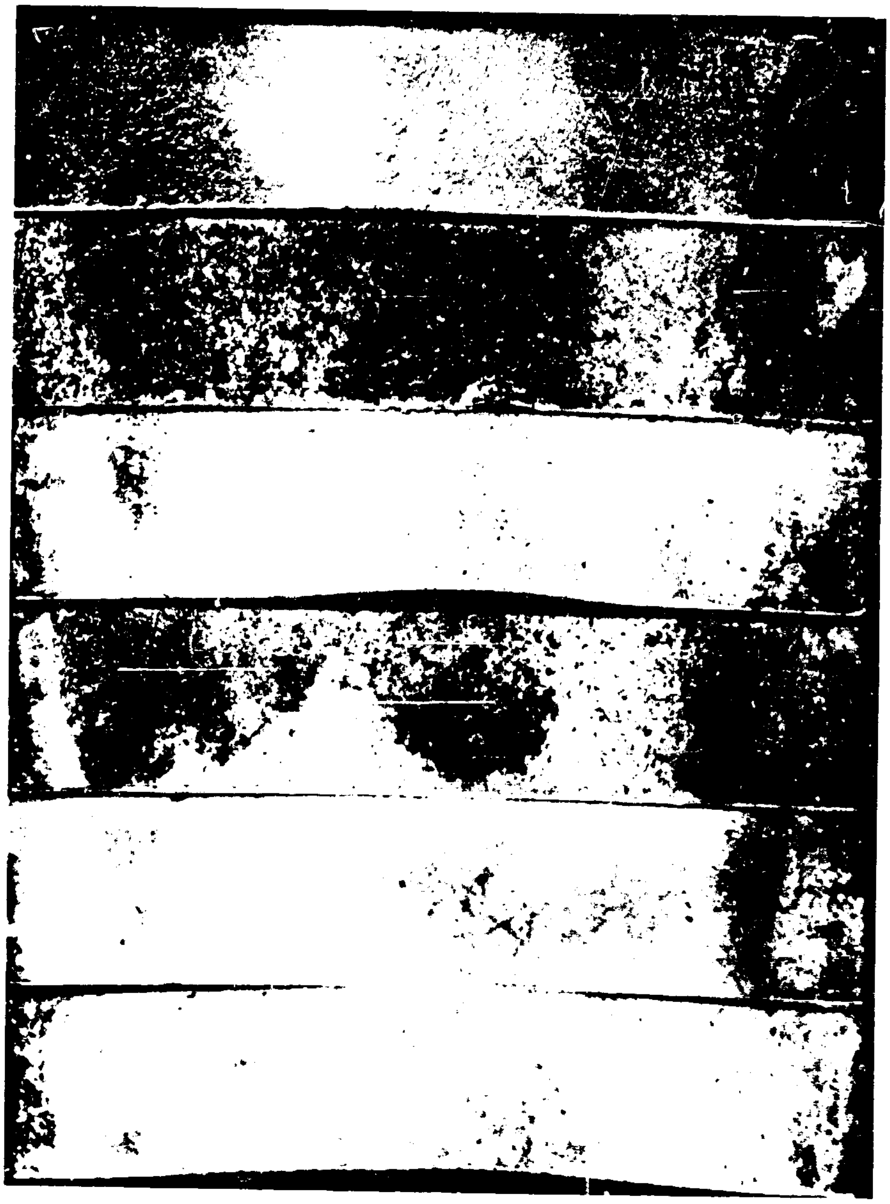


SULFUR IN FUEL, WT %	EXPOSURE TIME, HR
4.0	13
0.40	15
< 0.0040	15
4.0	10
0.40	10
< 0.0040	10

2X MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 7
MISCO MDC-1 COATED INCONEL 713C SPECIMENS FROM 2000 F TEST CONDITION WITH 10 PPM SEA SALT IN AIR

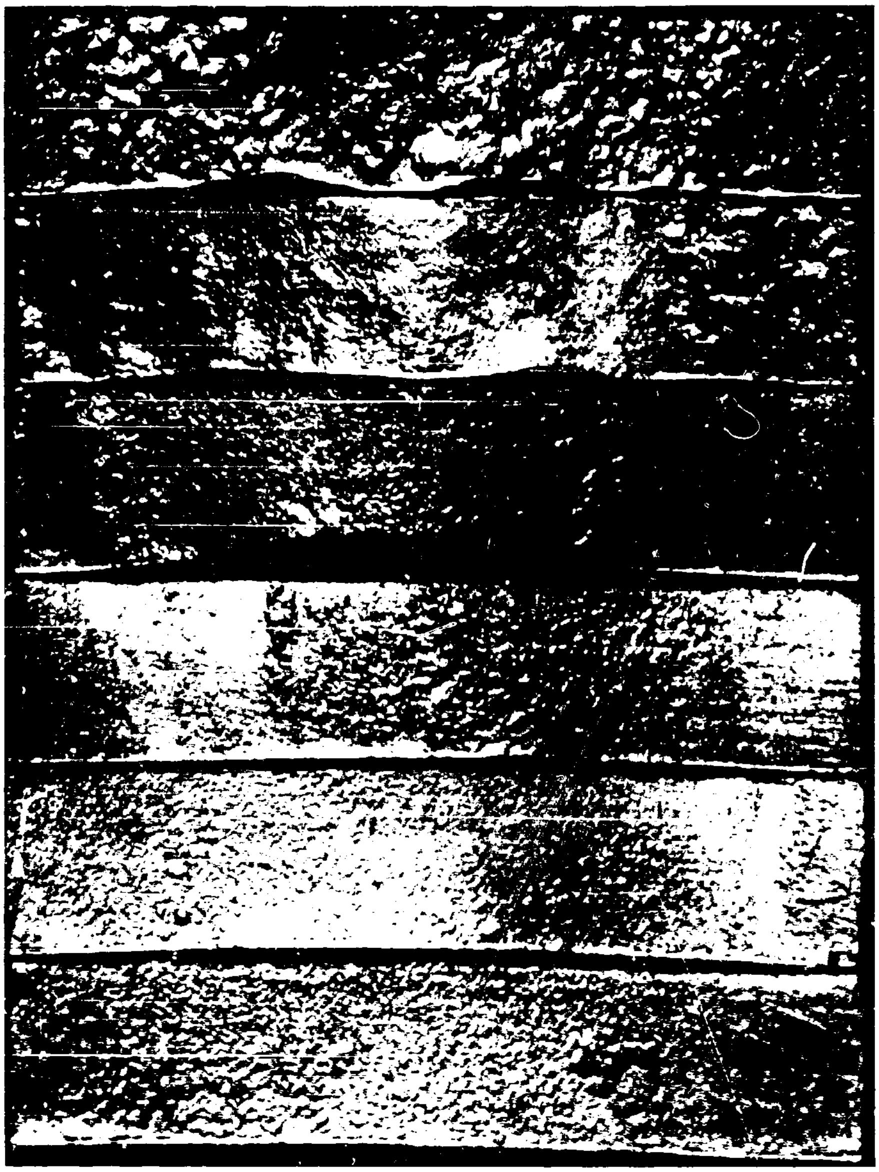
THIS ONE SPECIMEN WAS
ELECTRO–CLEANED TO SHOW
DEPOSIT–FREE SURFACE



SULFUR IN FUEL, WT %	SEA SALT IN AIR, PPM
0.40	1.0
0.040	1.0
<0.0040	1.0
0.40	NONE
0.040	NONE
<0.0040	NONE

2X MAGNIFICATION AFTER SONIC-CLEANING

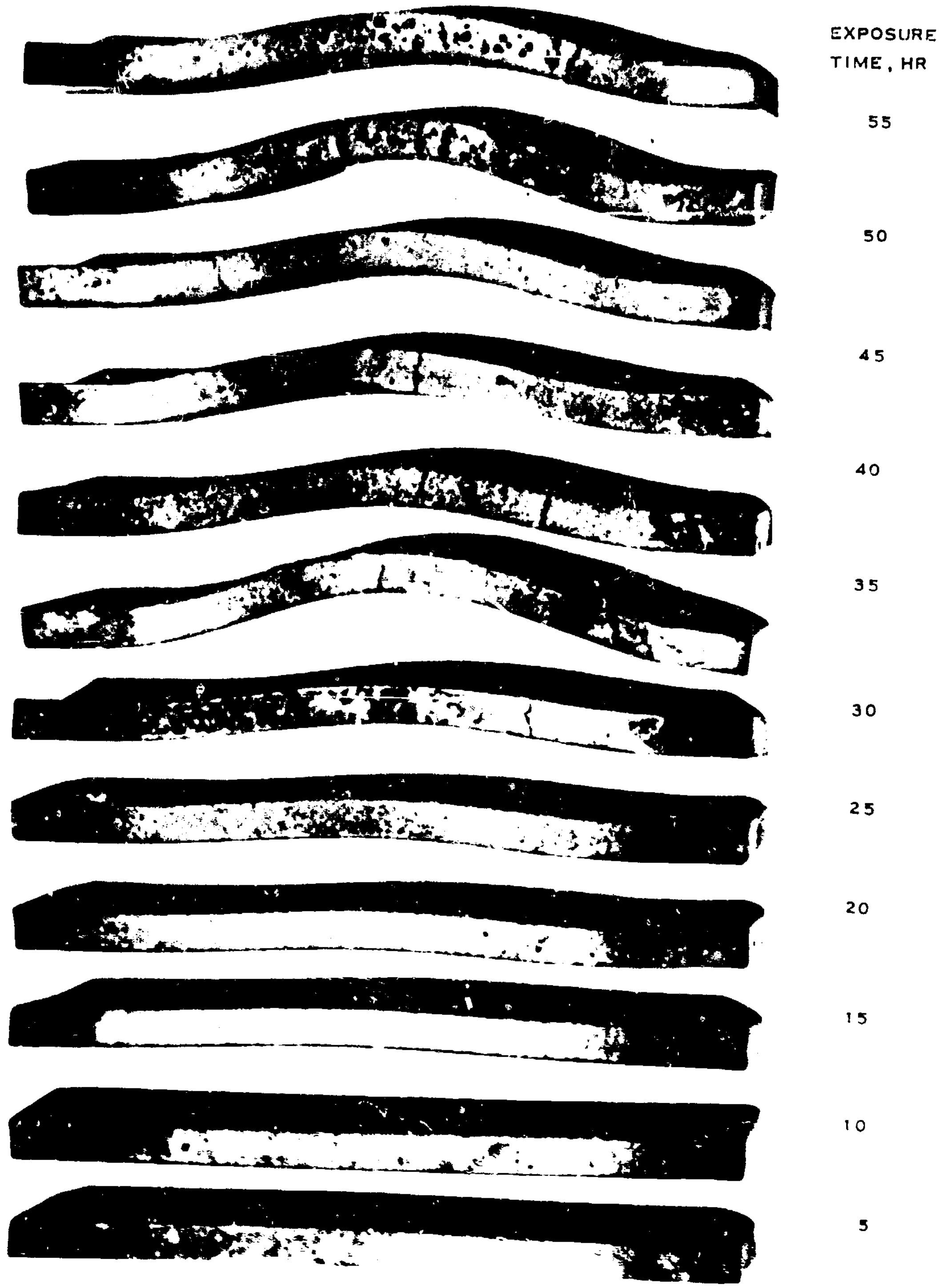
FIGURE 8
MISCO MDC-9 COATED INCONEL 713C SPECIMENS
AFTER 25 HOURS AT 2000 F TEST CONDITION



SULFUR IN FUEL, WT %	SEA SALT IN AIR, PPM
0.40	1.0
0.040	1.0
$<$ 0.0040	1.0
0.40	NONE
0.040	NONE
$<$ 0.0040	NONE

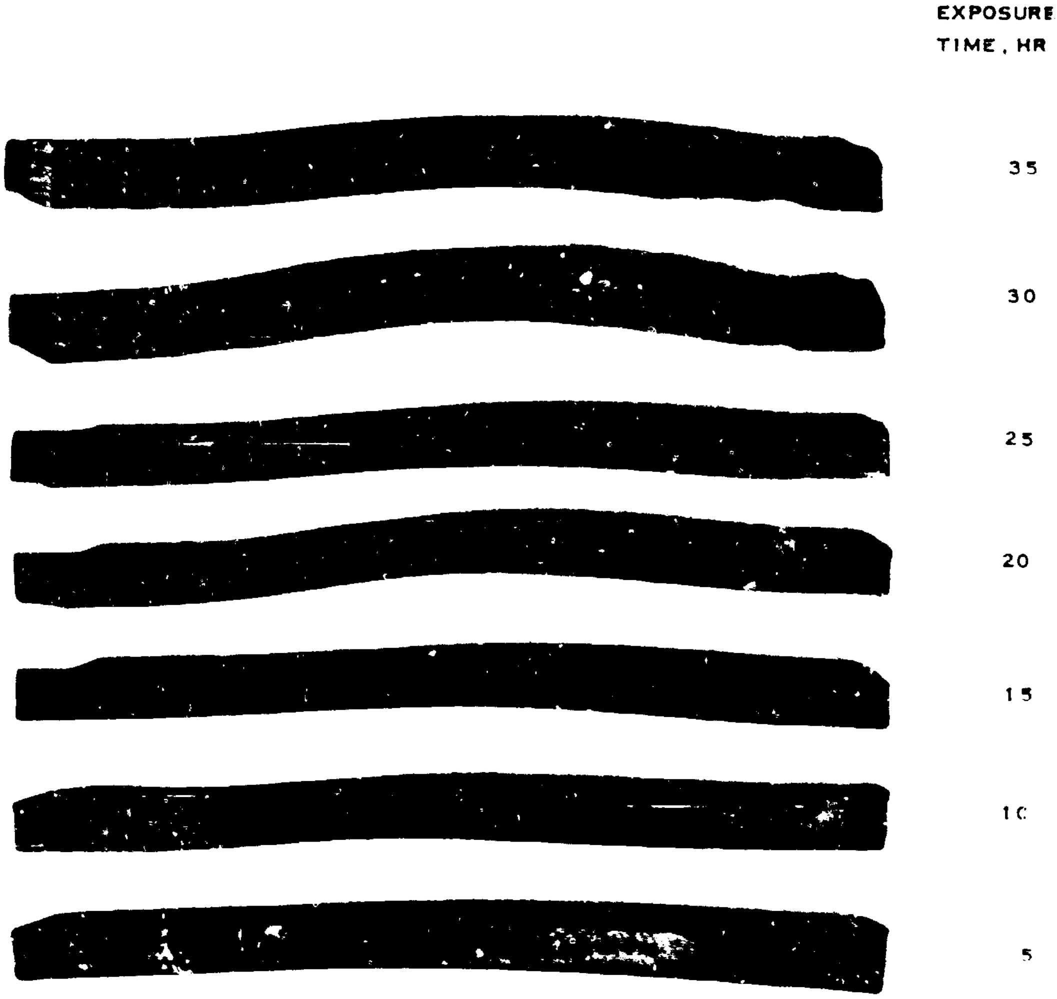
MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 9
INCONEL 713C SPECIMENS AFTER 25 HOURS
AT 2000 F TEST CONDITION



2X MAGNIFICATION OF SONICALLY CLEANED TEST SPECIMENS AFTER EXPOSURE IN PHILLIPS RIG AT 2000 F TEST CONDITION WITH NO SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL

FIGURE 10
DEVELOPMENT OF CRACKS IN LEADING EDGE
OF MISCO MDC-9 COATED INCONEL 713C SPECIMENS



2X MAGNIFICATION OF SONICALLY CLEANED TEST SPECIMENS
AFTER EXPOSURE IN PHILLIPS RIG AT 2000 F TEST CONDITION
WITH 1 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL

FIGURE 11
HOT CORROSION AND CRACKS IN LEADING EDGE
OF MISCO MDC-9 COATED INCONEL 713C SPECIMENS

The presence of cracks, observed without magnification, are noted in Tables 13 and 14 of Appendix 1 as part of the visual rating of specimens. These ratings are of the condition of the specimens at the time of their removal from test, which may not indicate the earliest time at which a specific specimen was cracked. While these ratings are inadequate for statistical analysis, an attempt has been made in Table 3 to show the effect of sulfur in fuel and "sea salt" in air on the extent of specimen cracking. This was done by listing the cumulative number of specimens removed from test at each time period in the test, along with the cumulative number of specimens that were cracked, and calculating the cumulative percentage cracked for the six combinations of sulfur in fuel and "sea salt" in air. It appears that the presence of 1.0 ppm "sea salt" in air increases the tendency of the specimens to crack, and increasing amounts of sulfur in the fuel tends to decrease the percentage of the specimens that are cracked.

4.2. Weight-Loss Data

In previous studies (1) of weight loss from hot corrosion, an estimate of the variance was found to be a percentage of the value, and logarithms of weight loss per unit area were used to provide a basis of uniform variance. The standard deviation of the weight-loss data in terms of logarithms (1) was 0.288, which is equivalent to a percentage standard deviation of 94 per cent. This information will aid in the evaluation of data from the current program.

In evaluating oxidation with time, many forms of equations have been proposed and used in the literature to express the relationship. These forms include linear, cubic, parabolic, and exponential equations depending upon the system being investigated. Plots of the data from the current investigation were made using linear coordinates and an example is shown in the lower portion of Figure 12 for Misco MDC-1 coated Inconel 713C exposed to 0.040 weight per cent sulfur in fuel and 1.0 ppm "sea salt" in air. The line is a visual fit of the data. The shape of the curve suggests the use of the exponential growth curve which is commonly used in the form.

$$W = (A)(B)^X, \tag{1}$$

where W is weight loss per unit area, X is exposure time, and A and B are constants to be evaluated. Applying logarithms to the equation,

$$\log W = \log A + (\log B)\ X \tag{2}$$

$$\text{or } Y = a + bX, \tag{3}$$

where $Y = \log W$, $a = \log A$, and $b = \log B$. Equation 3 is the equation for a straight line. The data in the upper portion of Figure 12 were fit using this form of equation and the calculated line is shown. The use of this form of equation permits the use of logarithms of weight

TABLE 3

SUMMARY OF CRACKS IN MISCO MDC-9 COATED INCONEL 713C TEST SPECIMENS

Total Exposure Time, Hours	Cumulative Totals					
	Zero Sea Salt in Air			1.0 ppm Sea Salt in Air		
	Specimens	Cracked	% Cracked	Specimens	Cracked	% Cracked
			≤ 0.0040 wt % Sulfur in Fuel			
5	1	0	0	1	0	0
10	2	0	0	2	1	50.0
15	3	0	0	3	2	66.7
20	4	1	25.0	5	3	60.0
25	5	2	40.0	7	5	71.4
30	6	2	33.3	8	6	75.0
35	7	3	42.8	9	7	77.8
40	8	4	50.0	10	8	80.0
45	9	5	55.6	11	9	81.8
50	10	6	60.0	-	-	-
55	11	7	63.6	-	-	-
			0.040 wt % Sulfur in Fuel			
5	1	0	0	1	1	100.0
10	2	0	0	3	2	66.7
15	3	0	0	5	3	60.0
20	4	0	0	7	4	57.1
25	5	1	20.0	9	6	66.7
30	6	2	33.3	10	7	70.0
35	7	3	42.9	11	8	72.7
40	8	4	50.0	-	-	-
45	9	5	55.6	-	-	-
50	10	6	60.0	-	-	-
55	11	7	63.6	-	-	-
			0.40 wt. % Sulfur in Fuel			
5	1	0	0	1	0	0
10	2	1	50.0	2	0	0
15	3	1	33.3	3	1	33.3
20	4	1	25.0	5	2	40.0
25	5	1	20.0	7	2	28.6
30	6	2	33.3	8	2	25.0
35	7	3	42.9	9	3	33.3
40	8	3	37.5	10	4	40.0
45	9	3	33.3	11	5	45.4
50	10	3	30.0	-	-	-
55	11	3	27.3	-	-	-

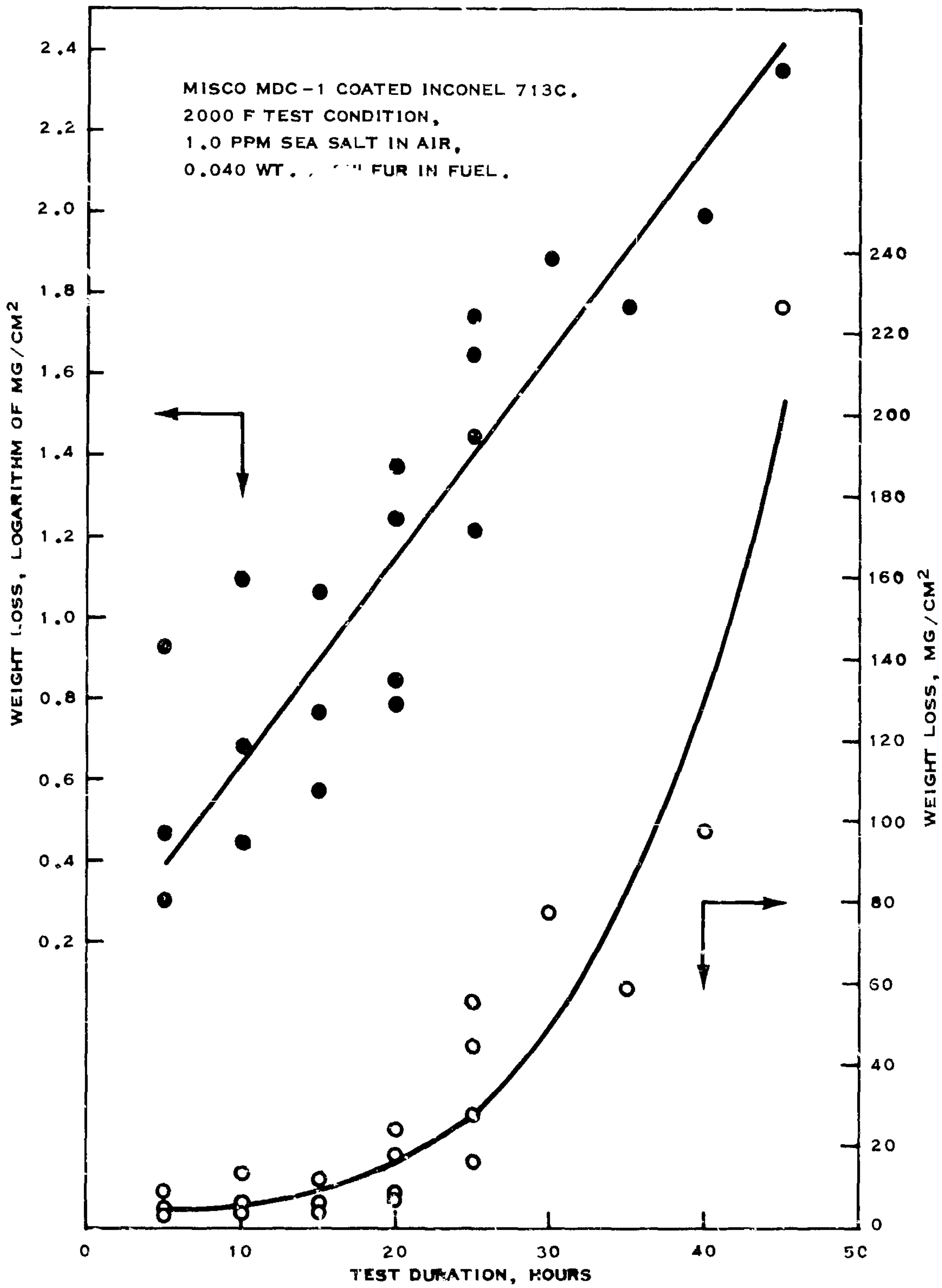


FIGURE 12
CURVE FITTING TO HOT CORROSION DATA

loss, which was shown previously to give uniform variance. Data that can be fit by Equation 3 shows that the logarithm of weight-loss increases at a uniform rate over the time-period studied. The slope, b, of Equation 3 is measured in terms of Δ logarithms per Δ hours, or the ratio of weight losses per hour [(mg/cm^2)/(mg/cm^2)/hour or (mg/cm^2)/hour/(mg/cm^2)]. Thus, b, is the relative rate of corrosion with units of reciprocal hours.

The data for each combination of sulfur in fuel and "sea salt" in air for the superalloy and the two coating-alloy systems shown in Tables 10 to 16 of Appendix 1 were fit to equations of the form of Equation 3. The constants, b, for the three equations with zero or 1.0 ppm "sea salt" describing the superalloy and the two coating-alloy systems, were compared by statistical "t" tests for significant differences at a 95 per cent confidence level. Where data for two or more levels of sulfur in fuel showed no statistically significant difference in the relative corrosion rates, at a given level of "sea salt" in air, the data were combined by fitting a single equation. In combining the data a sulfur term was included in the equation and the sulfur term was tested for statistical significance. If the sulfur term was not significant it was deleted and the equation was recalculated. The final equations which were calculated to represent the data are shown in Table 4. The data are also shown graphically in Figures 13 to 19, and the lines on the figures were calculated from the equations. Variability for the separate equations differ in magnitude, with eight of the equations having a S.E.E. of less than the standard deviation of 0.288 found in Reference 1, and five of the equations having a S.E.E. larger than the previous standard deviation. The size of the S.E.E. for most of the equations indicate that the variability remaining after removal of the effect of time is essentially the same as in the previous 5-hour tests on a variety of superalloys. With the small range of hours of exposure and the larger S.E.E. for the three equations for hot corrosion in the presence of 10.0 ppm "sea salt" in air, statistical comparisons are of doubtful value.

The equations for hot corrosion with time of exposure were calculated to establish statistically significant differences among concentrations of sulfur in fuel. The effect of sulfur in fuel on hot corrosion, with the level of "sea salt" in air and the test specimen material fixed, can be obtained from the equations.

Comparisons of the effect of sulfur in fuel on hot corrosion of Misco MDC-1 coated Inconel 713C can be made with zero and 1.0 ppm "sea salt" in air. Comparisons in the presence of 10.0 ppm "sea salt" in air will not be made because they are of doubtful value. In the absence of "sea salt" in air, a reduction in sulfur concentration, from the specification maximum of 0.40 weight per cent for JP-5 fuel to either 0.40 or $<$0.0040 weight per cent sulfur, significantly decreased the relative rate of corrosion; however, this may not be of practical significance because of the very low level of attack. In the presence of 1.0 ppm "sea salt" in air, a reduction in sulfur concentration from

TABLE 4

REGRESSION EQUATIONS FOR WEIGHT-LOSS VS TIME

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Regression Equations (a)	S.E.E. (b)	% S.E.E. (c)
		(Misco MDC-1 Coated Inconel 713C)		
< 0.0040	0.0	Y = 0.2478212 + 0.003211451 X	0.059775	14.7
0.040	0.0			
0.40	0.0	Y = 0.02214815 + 0.01390193 X	0.153822	42.5
< 0.0040	1.0	Y = 0.1883316 + 0.02158372 X	0.296952	98.2
0.040	1.0			
0.40	1.0	Y = 0.1377441 + 0.05072006 X	0.261855	82.8
< 0.0040	10.0	Y = 0.08116969 + 0.143218 X	0.404076	154
0.40	10.0	Y = -0.4523064 + 0.1520832 X	0.342303	119
4.0	10.0	Y = 0.0040024 + 0.08262738 X	0.398695	151
		(Misco MDC-9 Coated Inconel 713C)		
< 0.0040	0.0			
0.040	0.0	Y = -0.09654819 - 0.7288955 S + 0.0144935 X	0.154956	42.9
0.40	0.0			
< 0.0040	1.0	Y = -0.1946977 + 0.02867117 X	0.212553	63.2
0.040	1.0			
0.40	1.0	Y = -0.4717499 + 0.06061021 X	0.344121	121
		(Inconel 713C)		
< 0.0040	0.0			
0.040	0.0	Y = 0.8198599 + 0.02159729 X	0.153549	42.4
0.40	0.0			
< 0.0040	1.0	Y = 1.167399 + 0.02911474 X	0.198405	57.9
0.040	1.0			
0.40	1.0	Y = 1.012591 + 0.9080433 S + 0.048222 X	0.138587	37.6

(a) Y = a + cS + bX or Y = a + bX, where Y = logarithm of weight loss per unit area (mg/cm^2), S = weight per cent sulfur in fuel, and X = test duration, hours.

(b) Standard Error of Estimate in terms of logarithms.

(c) Standard Error of Estimate in terms of percentage of the value.

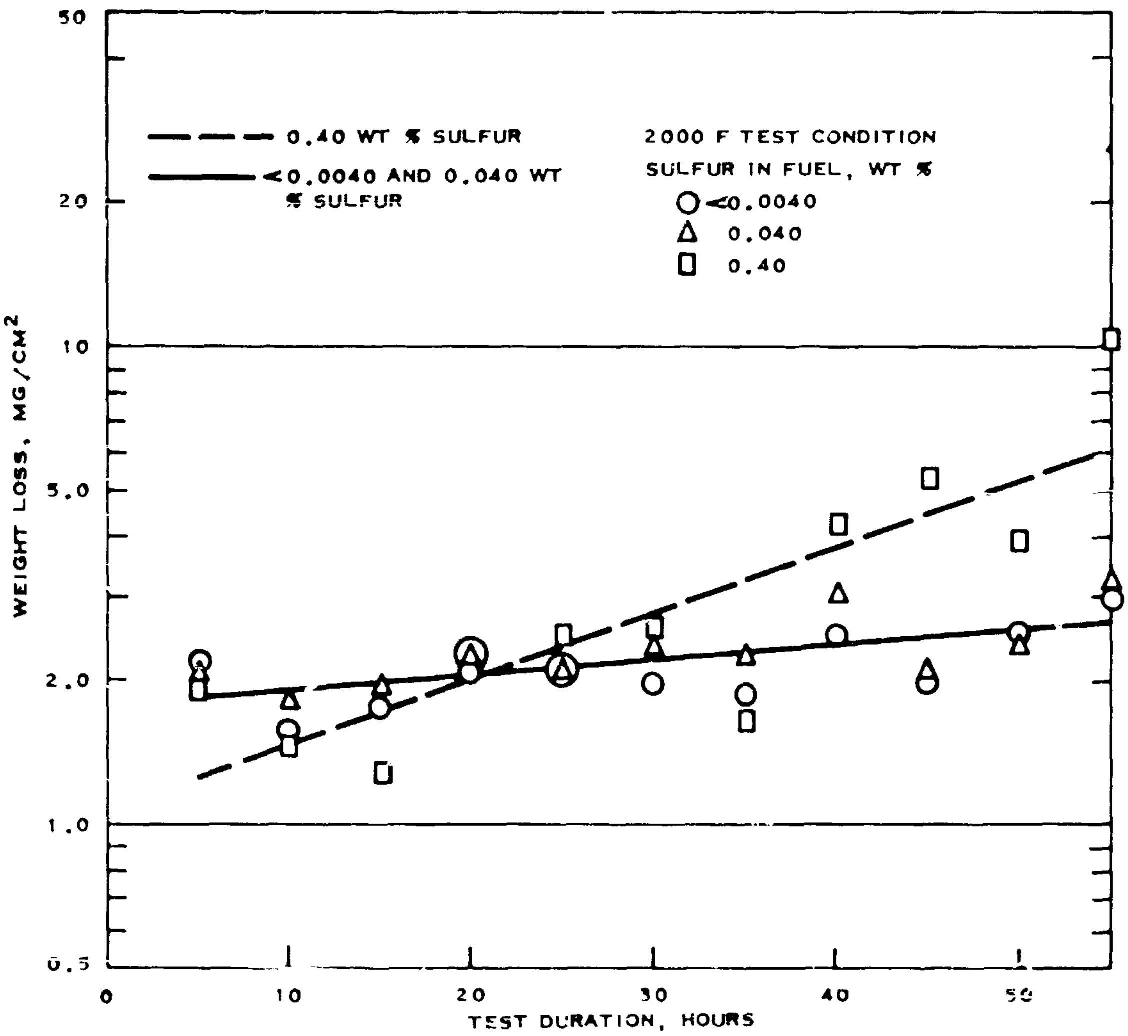


FIGURE 13
HOT CORROSION OF MISCO MDC-1 COATED INCONEL 713C WITH ZERO SEA SALT IN AIR

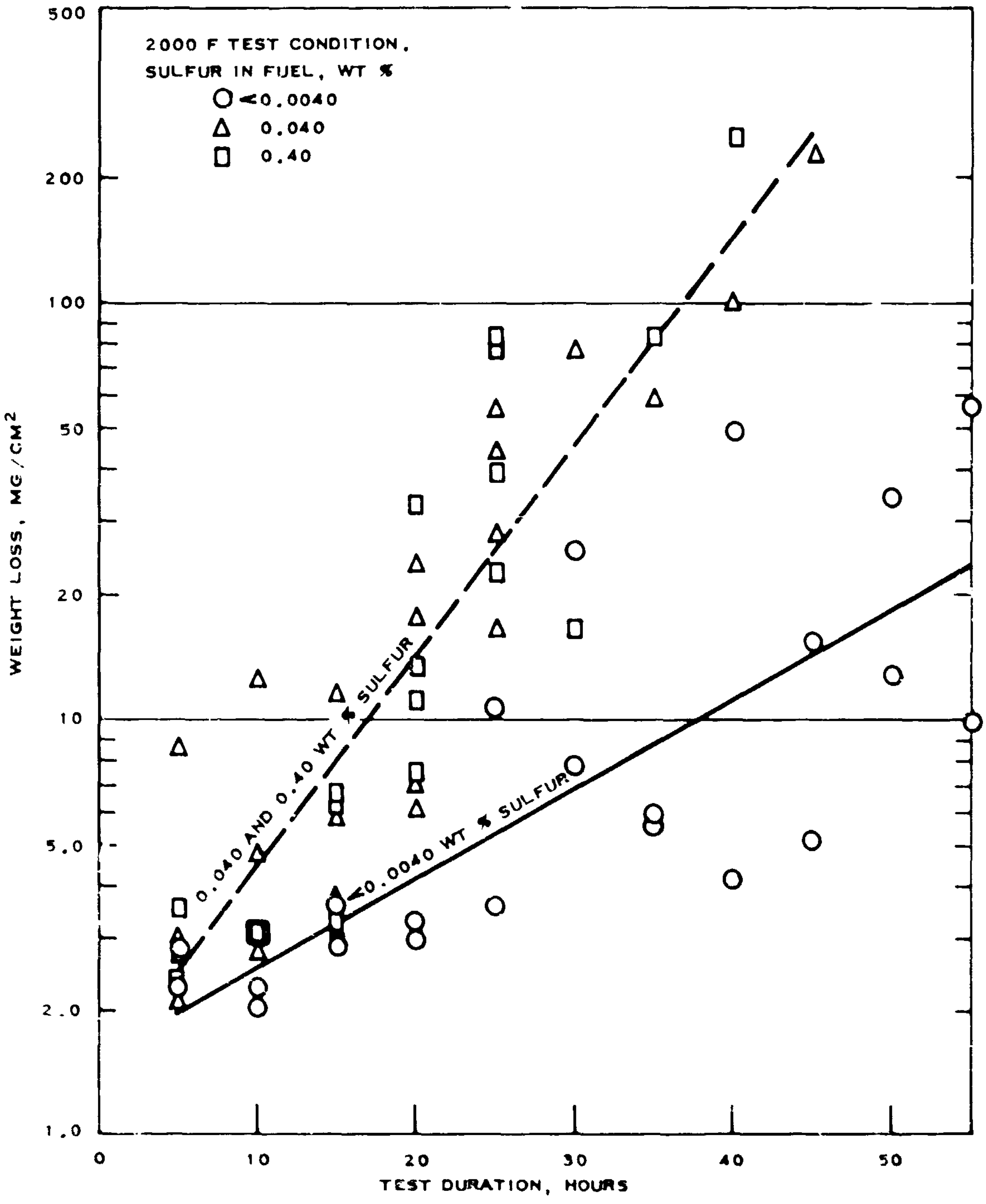


FIGURE 14
HOT CORROSION OF MISCO MDC-1 COATED INCONEL 713C WITH 1.0 PPM SEA SALT IN AIR

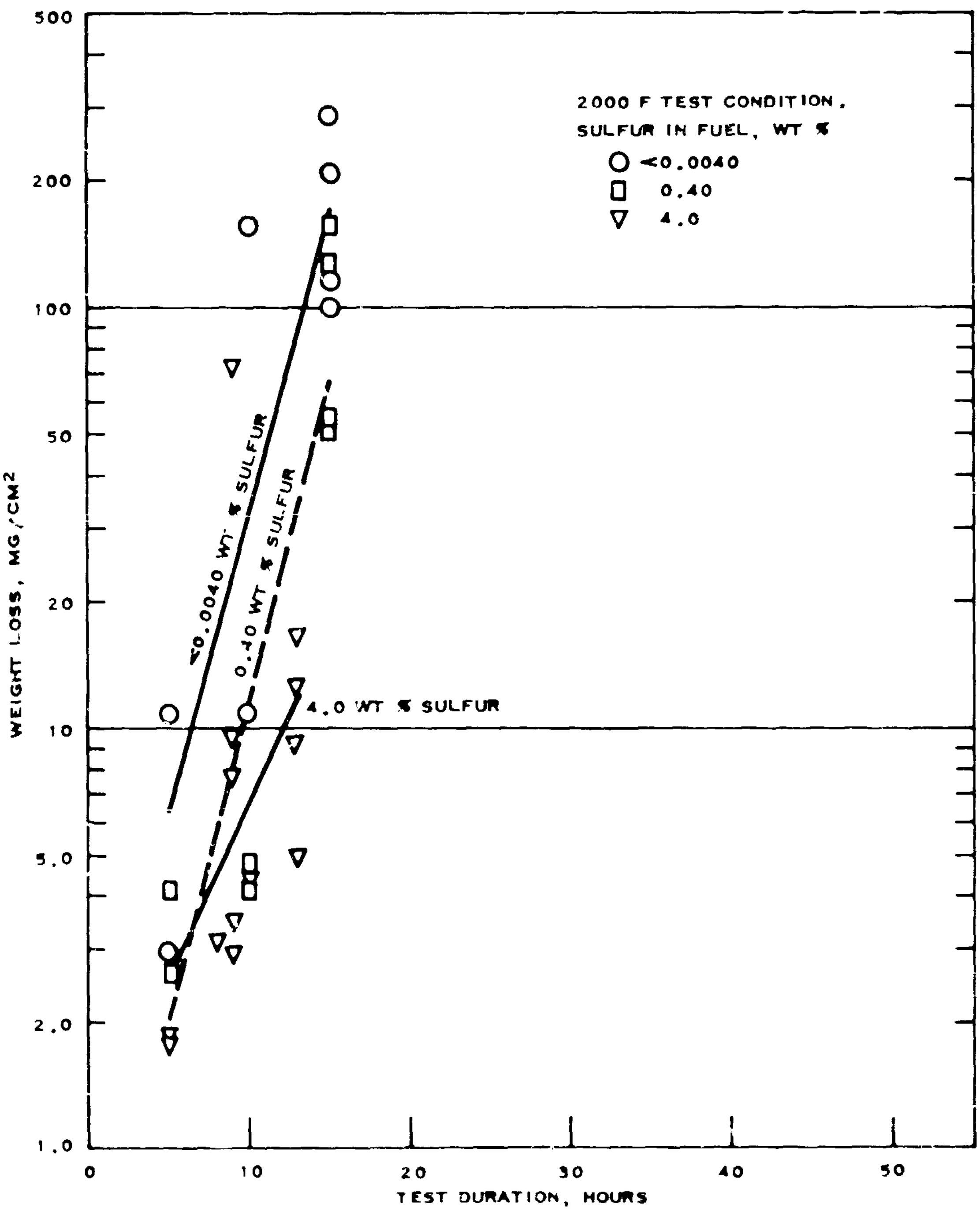


FIGURE 15
HOT CORROSION OF MISCO MDC-1 COATED INCONEL 713C WITH 10.0 PPM SEA SALT IN AIR

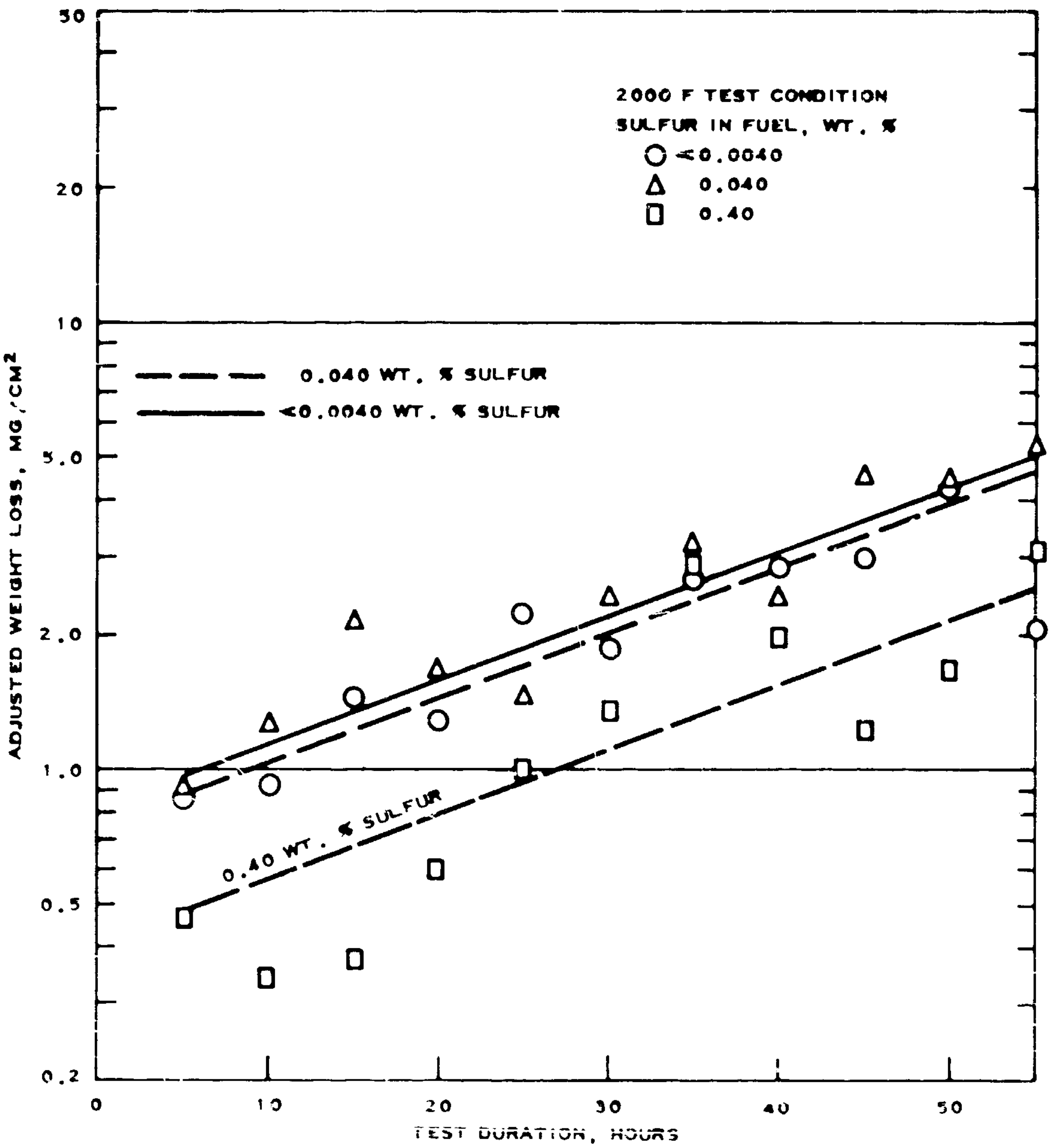


FIGURE 16
HOT CORROSION OF MISCO MDC-9 COATED INCONEL 713C WITH ZERO SEA SALT IN AIR

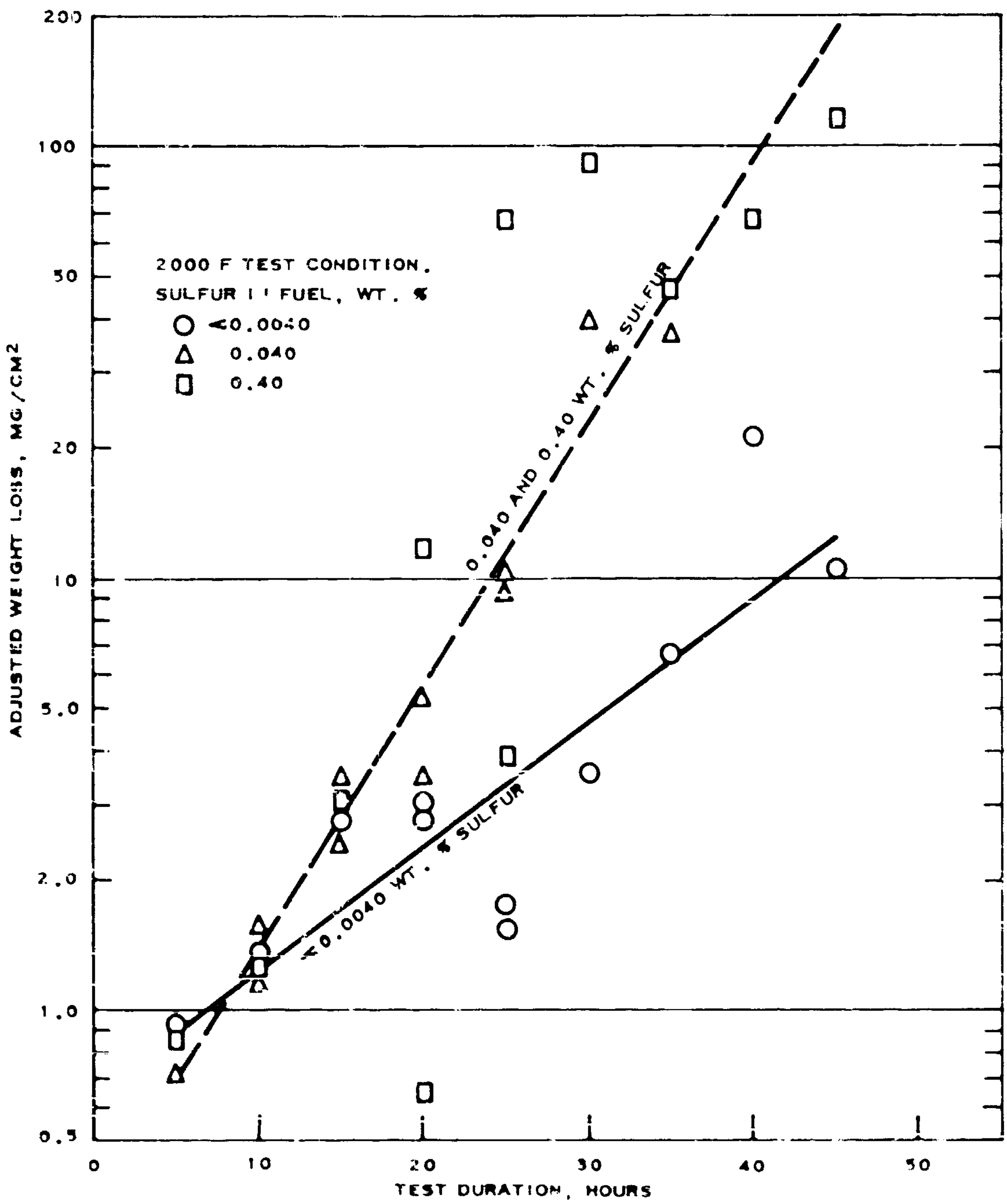


FIGURE 17
HOT CORROSION OF MISCO MDC-9 COATED INCONEL 713C WITH 1.0 PPM SEA SALT IN AIR

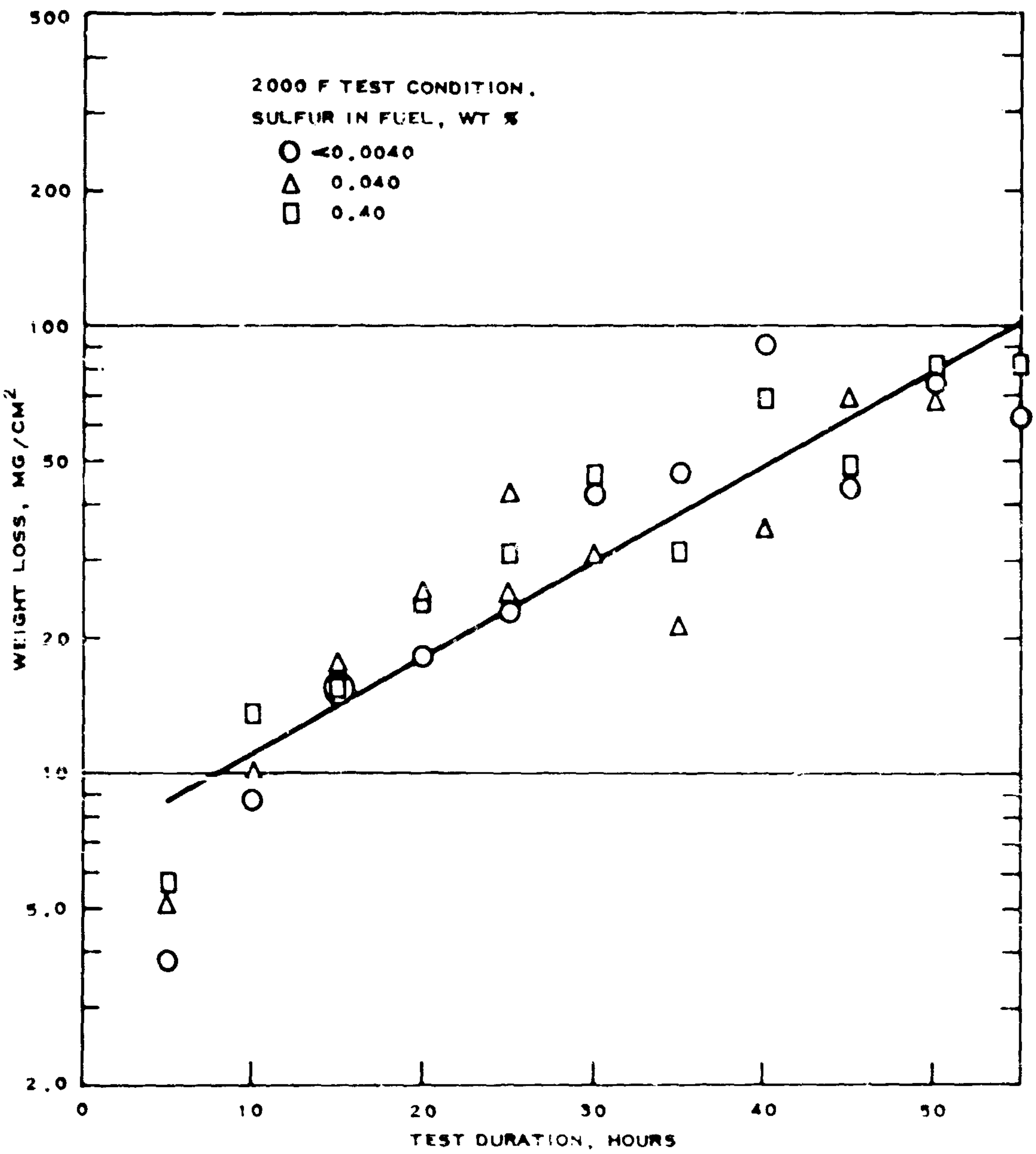


FIGURE 18
HOT CORROSION OF INCONEL 713C WITH ZERO SEA SALT IN AIR

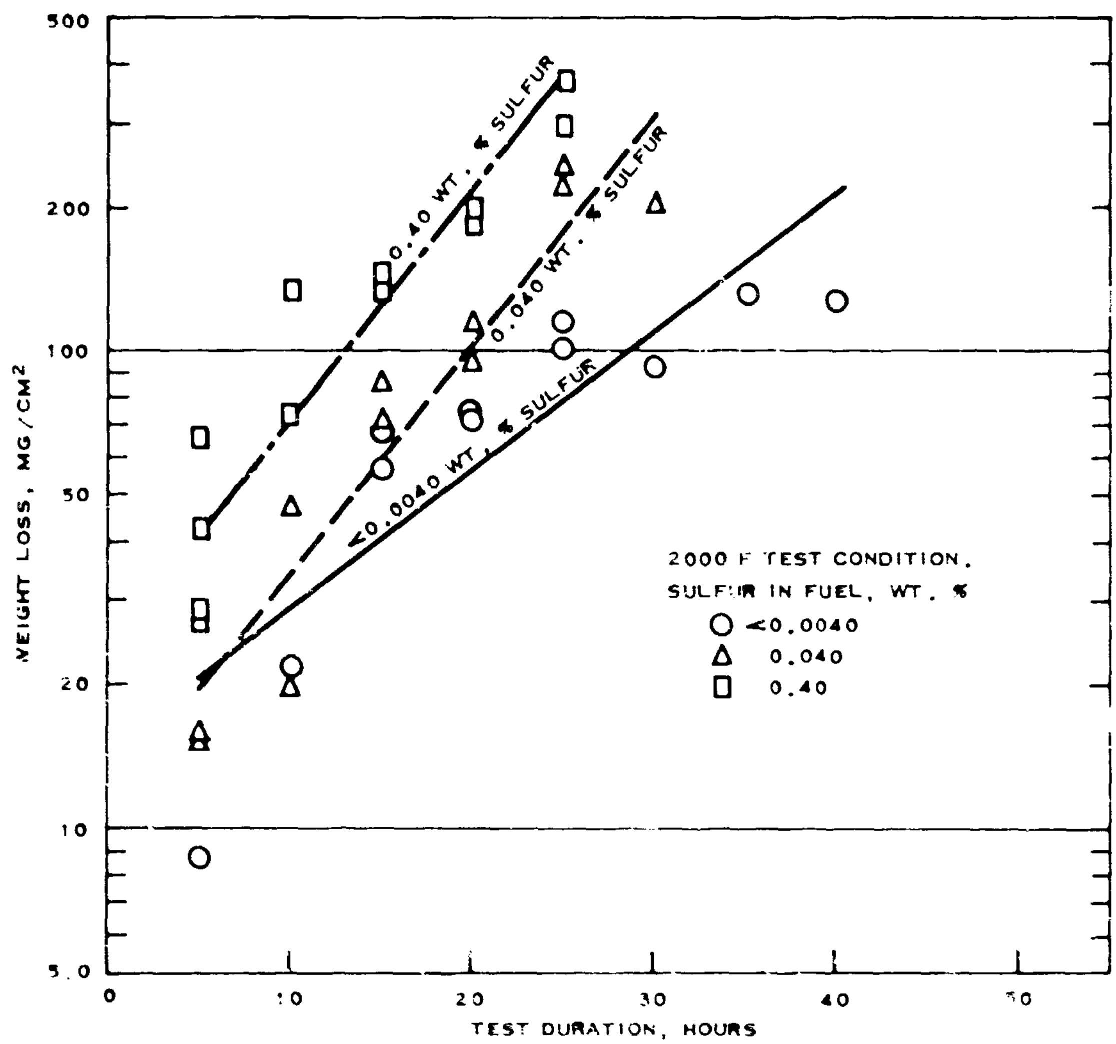


FIGURE 19
HOT CORROSION OF INCONEL 713C WITH 1.0 PPM SEA SALT IN AIR

the specification maximum of 0.40 to 0.040 weight per cent does not significantly change the relative corrosion rate; however, a reduction from 0.40 to <0.0040 weight per cent decreases the relative rate of corrosion significantly.

In comparisons of the effect of sulfur on hot corrosion of Misco MDC-9 coated Inconel 713C in the absence of "sea salt" in air, a single equation indicates that there are no significant differences in the relative rates of corrosion for the three concentrations of sulfur in the fuel. A sulfur term in the equation indicates a significant effect of fuel sulfur on the level of attack. Further testing shows that there is no statistically significant difference in the level of attack with <0.0040 or 0.040 weight per cent sulfur in fuel, but that it is less with 0.40 weight per cent sulfur. Thus, a reduction in sulfur concentration from the specification maximum of 0.40 to either 0.040 or <0.0040 weight per cent sulfur significantly increases hot corrosion; however, the differences may not be of practical significance because of the low level of attack. In the presence of 1.0 ppm "sea salt" in air, a single equation was obtained for Misco MDC-9 coated Inconel 713C with 0.40 and 0.040 weight per cent sulfur in fuel, and a second equation was obtained for data with <0.0040 weight per cent sulfur in fuel. These equations indicate no significant difference in the hot corrosion of Misco MDC-9 coated Inconel 713C with a reduction in fuel sulfur from the specification maximum of 0.40 to 0.040 weight per cent sulfur; however, a reduction in fuel sulfur from the specification maximum of 0.40 to <0.0040 weight per cent sulfur will significantly decrease hot corrosion.

A single equation was obtained with uncoated Inconel 713C specimens for three levels of sulfur in fuel, in the absence of "sea salt" in air, indicating no significant effect of the concentration of sulfur in fuel on the superalloy. In the presence of 1.0 ppm "sea salt" in air, a single equation was obtained for uncoated Inconel 713C with 0.40 and 0.040 weight per cent sulfur in fuel. This equation contained a sulfur term which indicates that the relative rates of corrosion for the two concentrations of sulfur in fuel are the same, but the levels of attack are significantly different. A second equation was obtained for data with <0.0040 weight per cent sulfur in fuel. From these equations it can be concluded that a reduction in sulfur concentration from the specification maximum of 0.40 weight per cent sulfur to either 0.040 or <0.0040 weight per cent sulfur will significantly decrease hot corrosion of Inconel 713C.

The relative corrosion rates and the 95 per cent confidence limits for these rates for each of the equations are shown in Table 5. In comparisons where the relative corrosion rate for one condition is not included in the confidence limits for another condition it can be concluded with 95 per cent confidence that the relative rates of corrosion are statistically different. If the confidence limits include zero, the relative rate of corrosion is not statistically significant.

TABLE 5

RELATIVE CORROSION RATES FROM REGRESSION EQUATIONS

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Relative Corrosion Rate (a)	95 % Confidence Limits Lower (a)	95 % Confidence Limits Upper (a)
		Misco MDC-1 Coated Inconel 713C		
< 0.0040	0.0	0.003211451	0.001530135	0.004892767
0.040	0.0			
0.40	0.0	0.01390193	0.00726687	0.02053699
< 0.0040	1.0	0.02158372	0.01312873	0.03003871
0.040	1.0			
0.40	1.0	0.05072006	0.04272244	0.05871768
< 0.0040	10.0	0.143218	0.0588950	0.2275410
0.400	10.0	0.1520832	0.0806510	0.2235154
4.0	10.0	0.08262738	-0.00837596	0.17365072
		Misco MDC-9 Coated Inconel 713C		
< 0.0040	0.0			
0.040	0.0	0.0144935	0.01100982	0.01797718
0.40	0.0			
< 0.0040	1.0	0.02867117	0.01635011	0.04099223
0.040	1.0			
0.40	1.0	0.06061021	0.04633117	0.07488925
		Inconel 713C		
< 0.0040	0.0			
0.040	0.0	0.02159729	0.01800603	0.02518855
0.40	0.0			
< 0.0040	1.0	0.02911474	0.01566623	0.04256325
0.040	1.0			
0.40	1.0	0.04822200	0.04041496	0.05602904

(a) Values and 95 per cent confidence limits for exponential equations in Table 4.

From the relative corrosion rates and confidence limits, comparisons can be made of the effect of "sea salt" in air with the sulfur concentration of the fuel and the test specimen material fixed.

Comparisons of relative corrosion rates for Misco MDC-1 coated Inconel 713C show that a reduction in "sea salt" in air from 10.0 to 1.0 ppm decreases the relative rate of hot corrosion and a reduction from 1.0 ppm to zero "sea salt" in air further decreases the relative corrosion rate.

All comparisons with Misco MDC-9 coated Inconel 713C show that a reduction from 1.0 ppm to zero "sea salt" in air decreases the relative corrosion rates.

With uncoated Inconel 713C a reduction in concentration of "sea salt" in air from 1.0 to zero decreased the relative corrosion rates in all comparisons.

Comparisons of the relative rates of corrosion for the two coatings with the relative corrosion rates for the bare alloy in the absence of "sea salt" in air, show that the coatings decrease hot corrosion. In the presence of 1.0 ppm "sea salt" in air, the relative corrosion rates for the two coatings are the same as for the bare alloy at the same level of sulfur in fuel.

Weight losses for 25 hours of exposure, which is near the midpoint of the experimental data, were calculated using each of the equations in Table 4. These data, which are shown in Table 6, provide a measurement of the level of attack on the two coatings and the bare alloy at the various combinations of sulfur in fuel and "sea salt" in air. In calculating the weight loss, a value of 25 was substituted for "X" in the equations to obtain weight loss in terms of logarithm, and the value was then converted by use of antilogarithms. The 95 per cent confidence intervals, in terms of logarithms, were calculated using the S.E.E. for the individual equations, and the confidence intervals were also converted by the use of antilogarithms. It is pertinent to note that the confidence interval is greater when using point estimates from the equations. As a result comparisons of effects that were declared statistically significant previously may no longer be significant. If the primary objective of our experiment had been to make point comparisons, data should have been concentrated at the point of interest; however, this was not the case. Never-the-less, comparisons that are shown to be statistically significant by points estimated from the equations are valid comparisons. Comparisons of the effects of sulfur in fuel and "sea salt" in air will not be made; however, significant effects are in agreement with previous analyses.

TABLE 6

PREDICTED WEIGHT LOSS FROM REGRESSION EQUATIONS
(25 Hours of Exposure)

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Predicted Weight Loss, mg/cm^2 (a)	95 % Confidence Interval on Weight Loss, mg/cm^2 (b) Lower	Upper
		Misco MDC-1 Coated Inconel 713C		
<0.0040	0.0	2.1	1.6	2.9
0.040	0.0	2.1	1.6	2.9
0.40	0.0	2.3	1.0	5.4
<0.0040	1.0	5.3	1.2	23
0.040	1.0	25	7.4	88
0.40	1.0	25	7.4	88
		Misco MDC-9 Coated Inconel 713C		
<0.0040	0.0	1.8	0.87	3.9
0.040	0.0	1.7	0.82	3.6
0.40	0.0	0.94	0.44	2.0
<0.0040	1.0	3.3	1.0	11
0.040	1.0	11	2.0	60
0.40	1.0	11	2.0	60
		Inconel 713C		
<0.0040	0.0	23	11	48
0.040	0.0	23	11	48
0.40	0.0	23	11	48
<0.0040	1.0	79	17	370
0.040	1.0	180	88	370
0.40	1.0	380	190	780

(a) Weight losses calculated by substitution of 25 for "X" in equations of Table 4 to obtain logarithms of weight losses and then converting to weight losses by use of antilogarithms.

(b) Confidence intervals calculated in terms of logarithms using the S.E.E. for individual equations and then converting to weight losses by use of antilogarithms.

From comparisons of data in Table 6, it can be concluded that the levels of attack on Misco MDC-1 and MDC-9 coated Inconel 713C specimens are less than with uncoated Inconel 713C specimens. It has been shown that the relative rates of corrosion for the two coatings are less than for the bare superalloy, in the absence of "sea salt" in air; however, in the presence of 1.0 ppm "sea salt" in air, the relative rates of corrosion for the coated and uncoated alloy were the same at the same level of sulfur in fuel. A possible explanation for the difference in level of attack with the same relative rates of corrosion may be that attack on the fresh, uncoated, Inconel 713C proceeds rapidly until a scale of corrosion products is established on the surface of the specimen, and attack then proceeds at the slower relative corrosion rates shown by the equations. Another possible explanation for the difference could be that the beneficial effect of the coating occurs during the early exposure of the specimens, and once the coating has been penetrated the relative rate of corrosion follows that of the bare alloy.

Comparisons of the "life" of the coating may be of interest to some of our readers. A measure of coating life can be obtained by selecting a criteria for failure based on weight loss and calculating hours to failure from the calculated equations. As already suggested, a criteria of 5.0 mg/cm^2 should be a realistic level for coating failure. Using this value as a criteria for coating failure, the hours to failure for the Misco MDC-1 and MDC-9 coated Inconel 713C at each of the combinations of sulfur in fuel and "sea salt" in air have been calculated from the equations of Table 4, and are shown in Table 7. Confidence limits have not been established for these values. Comparisons of the effect of sulfur in fuel and "sea salt" in air on life of the coatings will not be made, since they would be the same as drawn previously from relative rates of corrosion and levels of attack. One item of interest in Table 7 is that Misco MDC-1 coated Inconel 713C appears to be more affected by the combinations of sulfur in fuel and "sea salt" in air than does the Misco MDC-9 coated Inconel 713C; thus, for the three levels of sulfur in fuel, with either zero or 1.0 ppm "sea salt" in air, the range in hours to coating failure for the Misco MDC-1 coated Inconel 713C is 129 hours (from 140 to 1[illegible] hours), while for the Misco MDC-9 coated Inconel 713C it is 56 hours (from 75 to 19 hours).

In Reference 1, comparisons of the hot corrosion of five superalloys and a coated superalloy, in the presence of zero, 1.0 and and 10.0 ppm "sea salt" in air, were made for three levels of sulfur in fuel. These data have been summarized in Tables 8 and 9, and show the statistically significant effects of reducing sulfur in fuel from the specification maximum for grade JP-5 of 0.40 to 0.040 and from 0.40 to $<$0.0040 weight per cent. Data from the current program also have been included in these tables, where available. In these tables a statistically significant decrease in hot corrosion with a reduction of sulfur in fuel is indicated by plus (+), no statistically significant change by zero (0), and a statistically significant increase in hot corrosion with a reduction of fuel sulfur by minus (-).

TABLE 7

LIFE OF COATING ON INCONEL 713C
(Failure Criterion = 5.0 mg/cm^2 Weight Loss)

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Time (a) to Failure, hours
	Misco MDC-1	
< 0.0040	0.0	140
0.040	0.0	140
0.40	0.0	49
< 0.0040	1.0	24
0.040	1.0	11
0.40	1.0	11
< 0.0040	10.0	4
0.40	10.0	8
4.0	10.0	8 ?
	Misco MDC-9	
< 0.0040	0.0	55
0.040	0.0	57
0.40	0.0	75
< 0.0040	1.0	31
0.040	1.0	19
0.40	1.0	19

(a) Calculated from equations of Table 4.

TABLE 8

EFFECT OF REDUCING SULFUR FROM 0.40 TO 0.040 WT % IN JP-5 ON HOT CORROSION

Sea Salt in Air, ppm	Specimen Temp., F	Superalloys (a) WI-52	(a) U-500	(a) IN-100	(a) SM-200	(b) Inconel 713C	(c) MDC-1 Coated 713C	(c) MDC-9 Coated 713C
0.0	1375	+	+	+	+	+	...	...
0.0	1537	+	0	0	+	+	...	...
0.0	1814	0	0	0	0	0	...	...
0.0	1970	0	0	0	0	0 0	+	-
0.0	2124	0	0	0	0	0	...	...
1.0	1375	+	+	+	+	+	...	...
1.0	1537	+	0	+	+	+	...	...
1.0	1814	0	0	0	0	0	...	...
1.0	1970	0	0	0	0	0 +	0	0
1.0	2124	0	0	0	0	0	...	...
10.0	1375	0	0	0	+	+	...	...
10.0	1537	0	-	-	+	+	...	...
10.0	1814	0	0	-	-	0	...	...
10.0	1970	0	0	0	-	0	...	...
10.0	2124	0	0	0	-	0	...	...

Comparisons of specimen weight-loss at 95 per cent confidence level.

+ = Attack decreased with sulfur reduction
− = Attack increased with sulfur reduction
0 = No change with sulfur reduction

(a) Data from Reference 1.

(b) First value from Reference 1 and second value from current program.

(c) Data from current program.

TABLE 9

EFFECT OF REDUCING SULFUR FROM 0.40 TO <0.004 WT %

IN JP-5 ON HOT CORROSION

Sea Salt in Air, ppm	Specimen Temp., F	Superalloys (a) WI-52	(a) U-500	(a) IN-100	(a) SM-200	(b) Inconel 713C	(c) MDC-1 Coated 713C	(c) MDC-9 Coated 713C
0.0	1375	+	+	+	+	+	...	...
0.0	1537	+	0	0	+	0	...	...
0.0	1814	0	0	0	0	+	...	...
0.0	1970	0	0	0	0	+ 0	+	-
0.0	2124	0	0	+	+	+	...	...
1.0	1375	+	+	0	+	+	...	...
1.0	1537	+	0	0	+	0	...	...
1.0	1814	0	0	+	0	+	...	...
1.0	1970	0	0	+	0	+ +	+	+
1.0	2124	0	0	+	0	+	...	...
10.0	1375	0	0	+	+	+	...	...
10.0	1537	0	-	0	+	0	...	...
10.0	1814	0	0	-	-	+	...	...
10.0	1970	0	0	0	-	+	...	...
10.0	2124	0	0	0	-	+	...	...

Comparisons of specimen weight-loss at 95 per cent confidence level.
+ = Attack decreased with sulfur reduction
- = Attack increased with sulfur reduction
0 = No change with sulfur reduction

(a) Data from Reference 1.

(b) First value from Reference 1 and second value from current program.

(c) Data from current program.

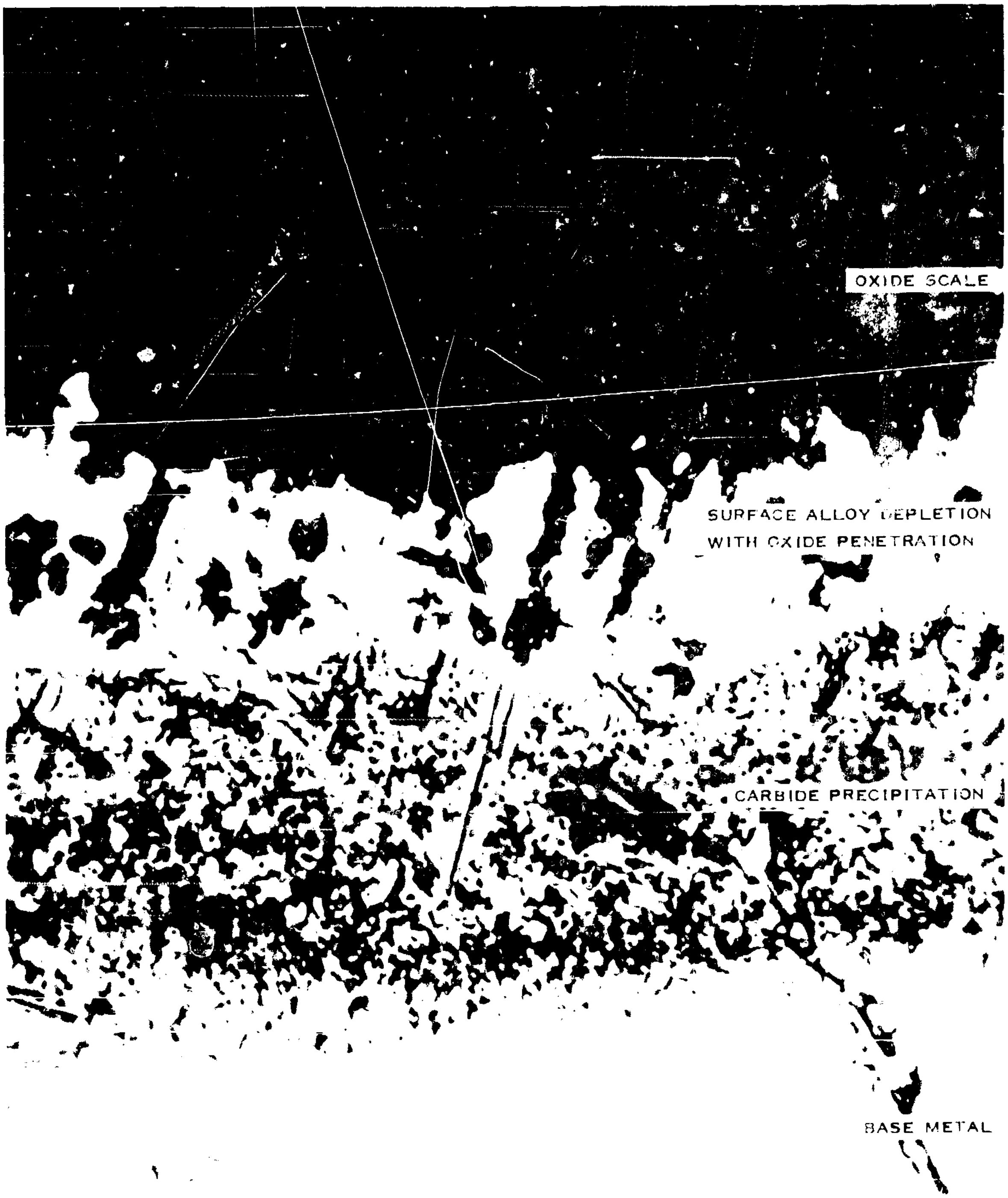
Comparing the effects of reducing sulfur in fuel from 0.40 to 0.040 per cent by weight, shown in Table 8, it can be seen that they vary with temperature, superalloy, and concentration of "sea salt" in air. Current data for Misco MDC-1 and MDC-9 coated Inconel 713C, and uncoated Inconel 713C, do not indicate the need for a change in the previous recommendation; that no reduction in the sulfur limit for grade JP-5 fuel, from 0.40 to 0.040 weight per cent, be made to alleviate hot-corrosion attack on turbine blades.

Comparing the effects of reducing sulfur in fuel from 0.40 to $<$0.0040 per cent by weight, shown in Table 9, it can be seen that they also vary with temperature, superalloy, and concentration of "sea salt" in air. Both previous and current data, in the presence of either zero or 1.0 ppm "sea salt" in air show that this larger reduction in fuel sulfur significantly decreases hot-corrosion in a greater number of cases. In only one instance (Misco MDC-9 coated Inconel 713C) does such removal of sulfur from the fuel increase hot corrosion, and in that case the level of attack was low at both sulfur concentrations. In the presence of 10.0 ppm "sea salt" in air, the reduction in sulfur decreases hot-corrosion for some alloys and conditions, and increases hot-corrosion for others. From these data, it appears that a reduction in the specification maximum for grade JP-5, from 0.40 to $<$ 0.0040 weight per cent sulfur, could decrease hot corrosion in a light "sea salt" environment, but could have a detrimental effect on hot-corrosion in a heavy "sea salt" environment.

4.3. Metallography

Information concerning the mode and the intensity of corrosive attack, sustained by the coatings and superalloy over the range in conditions of exposure, is pertinent to this investigation. High levels of specimen weight-loss are obviously objectionable; but lower values do not establish a lack of metal damage, for the attack may have penetrated the metal matrix by deep intercrystalline corrosion without significant metal loss. Therefore, metallographic examination of selected test specimens was made to evaluate the validity of our use of weight-loss data during this investigation.

A typical example of the oxidation structure at the surface of a turbine blade after long-time exposure is shown in Figure 20. This photomicrograph was taken at the convex surface of a cross-section from the center of a first-stage turbine blade which had completed 1800 hours in domestic-airline service since overhaul. An oxide scale covers the surface to a depth of about 0.001 inch. Alloy depletion, with some intergranmular oxidation, precedes surface oxidation by about 0.001 inch. This is attributed to the diffusion of more reactive constituents, such as chromium and aluminum, to the surface where they combine with oxygen to form the protective scale.



KALLING'S REAGENT ETCH, 2000X MAGNIFICATION.

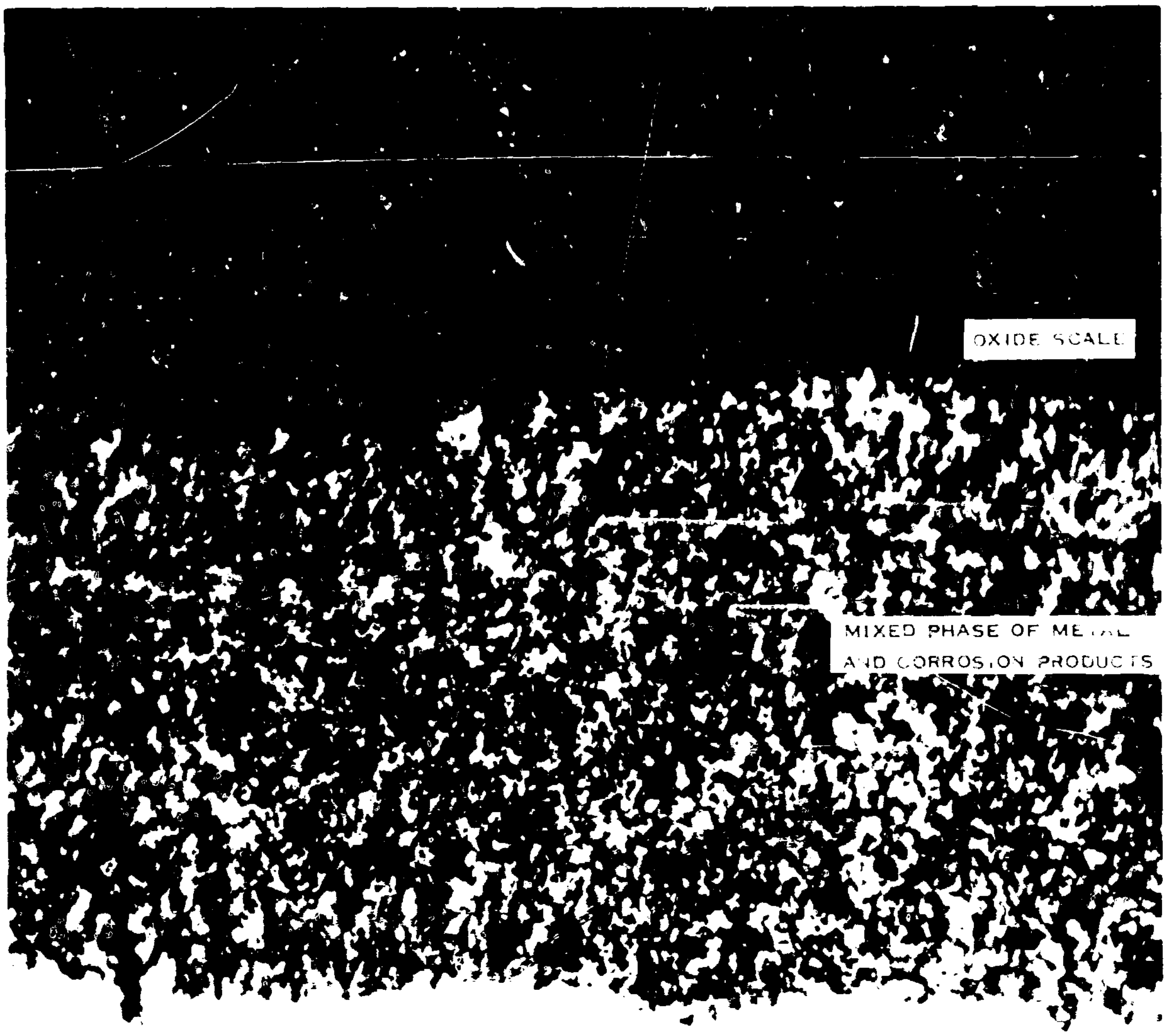
FIGURE 20
OXIDATION OF UDIMET 500 TURBINE BLADE

The catastrophic nature of hot corrosion, experienced by aviation-turbine engines when operated in a marine environment, is shown in Figure 21. This photomicrograph was taken at the leading edge of a cross-section from the center of a first-stage turbine blade recovered from a scrap drum at a U. S. Navy engine-overhaul shop. There is deep sub-surface oxidation, which produces a mixed-phase zone composed of metal and corrosion products; however, considerably metal loss can be sustained without blade failure because the attack advances on a broad front without deep-intercrystalline penetration of sulfides or oxides.

Features of hot corrosion at the interface between the oxides and the base matrix are shown in Figure 22, which is a further magnification of the section already shown in Figure 21. The attack is led by penetration of randomly-dispersed, light-grey, globules of metallic sulfides. Formation of these sulfides is associated with changes in the surface composition of the alloy, which is characterized by chromium depletion. Rapid oxidation of the weakened layer of alloy follows sulfide penetration. The mechanism of hot corrosion is complex and not fully understood.

Following exposure, test specimens from our investigation were cleaned, using techniques described in Appendix 2, and their weight loss was determined. Then specimens were chosen from each test for metallographic examination. The selection was made to obtain specimens with varying exposure time from each test, to allow study of any changes in the mode and intensity of corrosive attack. This included Misco MDC-1 coated Inconel 713C specimens which had been exposed for 10, 20, and 55 hours, if available, at nine test conditions; Misco MDC-9 coated Inconel 713C specimens which had been exposed for 20, 35, and 55 hours, if available, at six test conditions; and for reference the uncoated Inconel 713C specimens with maximum time of exposure, 25 to 55 hours, at six test conditions.

These test specimens were sectioned at the point of maximum-visible attack, mounted in Bakelite, and polished using procedures previously described (1). In general, the attack was most evident on the impact surface of the specimens, as expected, because it had maximum exposure to the corrosive agents. Photomicrographs showing typical attack on the specimens were taken there at both 200X and 1500X, and in some cases at other magnifications. Also, the coupons were frequently etched to show areas of alloy depletion and grain structure. It is considered neither necessary nor desirable to reproduce in this report the several hundred photomicrographs taken during our metallographic examination. Rather, typical photomicrographs are presented to illustrate the nature of the attack experienced by the superalloy and coatings over the range of conditions investigated.



UNETCHED 400X MAGNIFICATION

FIGURE 21
HOT CORROSION OF INCONEL 713C TURBINE BLADE

MARBLE'S REAGENT ETCH, 4000X MAGNIFICATION

FIGURE 22
HOT CORROSION OF INCONEL 713C TURBINE BLADE

The surface and matrix of an uncoated Inconel 713C specimen, in the as-received condition from Misco and as placed in test, is shown in Figure 23. This photomicrograph is included for reference purposes to emphasize that prior to exposure the test specimens were free from any unusual surface scale or impurities. The uniformity of the etch, to the very edge of the coupon, indicates that the surface alloy was not depleted.

Exposure of uncoated Inconel 713C in an environment free of "sea salt" resulted in only a slight, uniform surface oxidation. This was preceded by a limited zone of alloy depletion, which increased in depth with increasing exposure time. The addition of sulfur, via the fuel, to this environment (2000 F) did not result in the formation of metal sulfides at the corrosion interface, nor in accelerated oxidation attack on the depleted surface alloy, as illustrated by Figure 24. The similarity between the micro-features of oxidation attack evident on turbine blades from service engines and that on specimens exposed in Phillips test rig can be seen by comparison with Figure 20. When making this comparison, it should be remembered that the heavy surface scale has been removed from the test specimen by electro-cleaning.

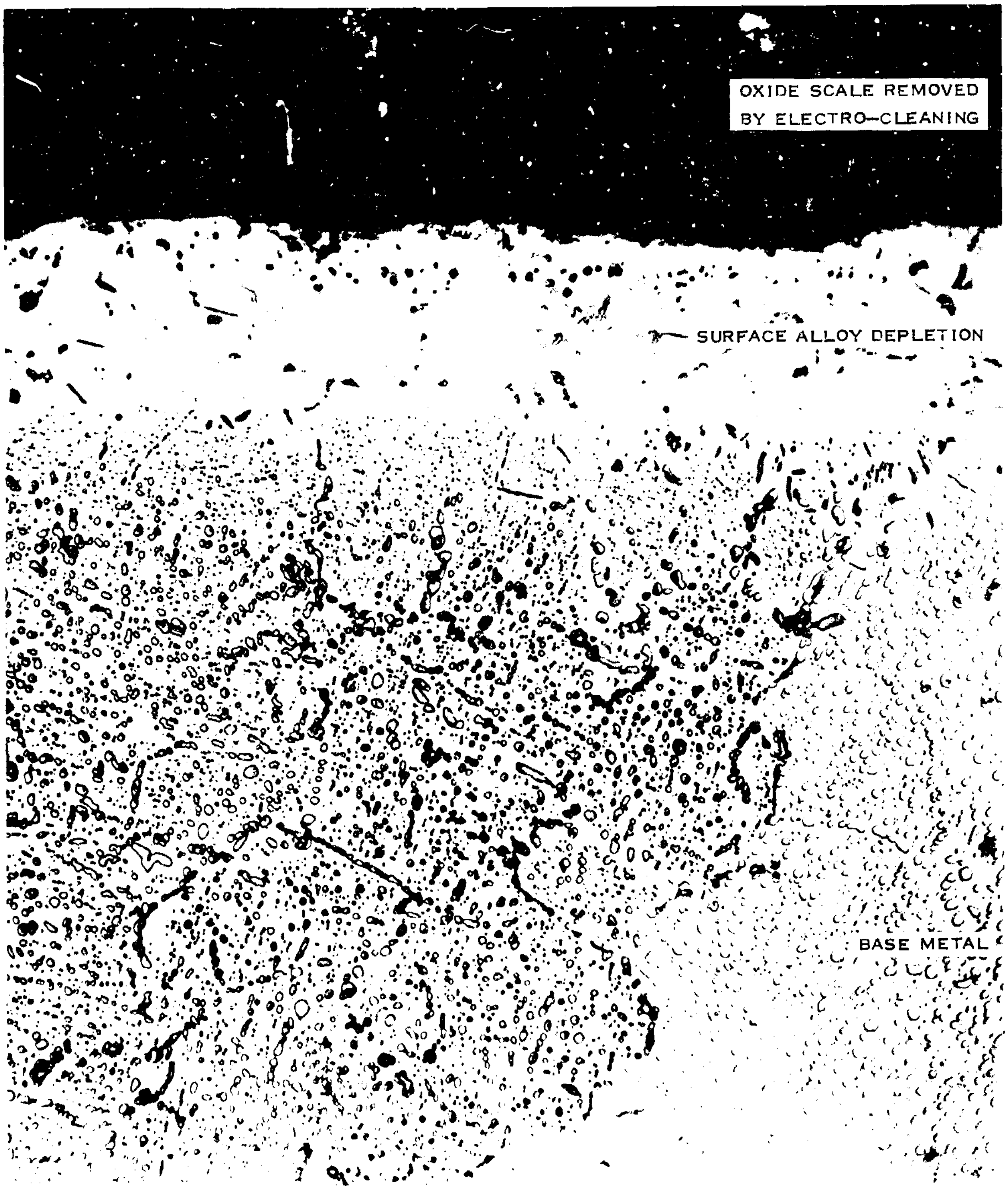
With the addition of "sea salt", via the air, to this environment, characteristic hot corrosion was encountered on uncoated Inconel 713C. Only the extent of the attack increased with increasing exposure time. The further addition of sulfur, via the fuel, to this "sea salt" environment affected neither the mode nor the intensity of the attack, which is shown in Figure 25. The micro-features of hot corrosion on Inconel 713C specimens exposed in Phillips test rig are quite similar to those evident on Inconel 713C turbine blades from service engines, which can be seen by comparison with Figure 22.

The surface and matrix of a typical MDC-1 coated Inconel 713C specimen, in the as-received condition from Misco and as placed in test, is shown in Figure 26. The aluminum-type coating was applied to obtain a total depth of approximately 2 mils; which is divided about equally between an outer-layer with non-metallic dispersions and a diffusion-layer. Previous experience (1) has shown that the electro-cleaning procedure, detailed in Appendix 2, does not alter the appearance of this coating.

In those areas where the MDC-1 coating remained intact following exposure, evidence was found of alloy depletion in the diffused layer, as shown in Figure 27. This modification of the coating remnant became increasingly evident with increasing exposure time, but was not favored by any other exposure variable in this probram. Similar coating depletion was experienced with extended exposure of Misco MDC-1 coated Inconel 713C specimens during exploratory tests (2).

MARBLE'S REAGENT ETCH. 1000X MAGNIFICATION.

FIGURE 23
UNEXPOSED INCONEL 713C SPECIMEN

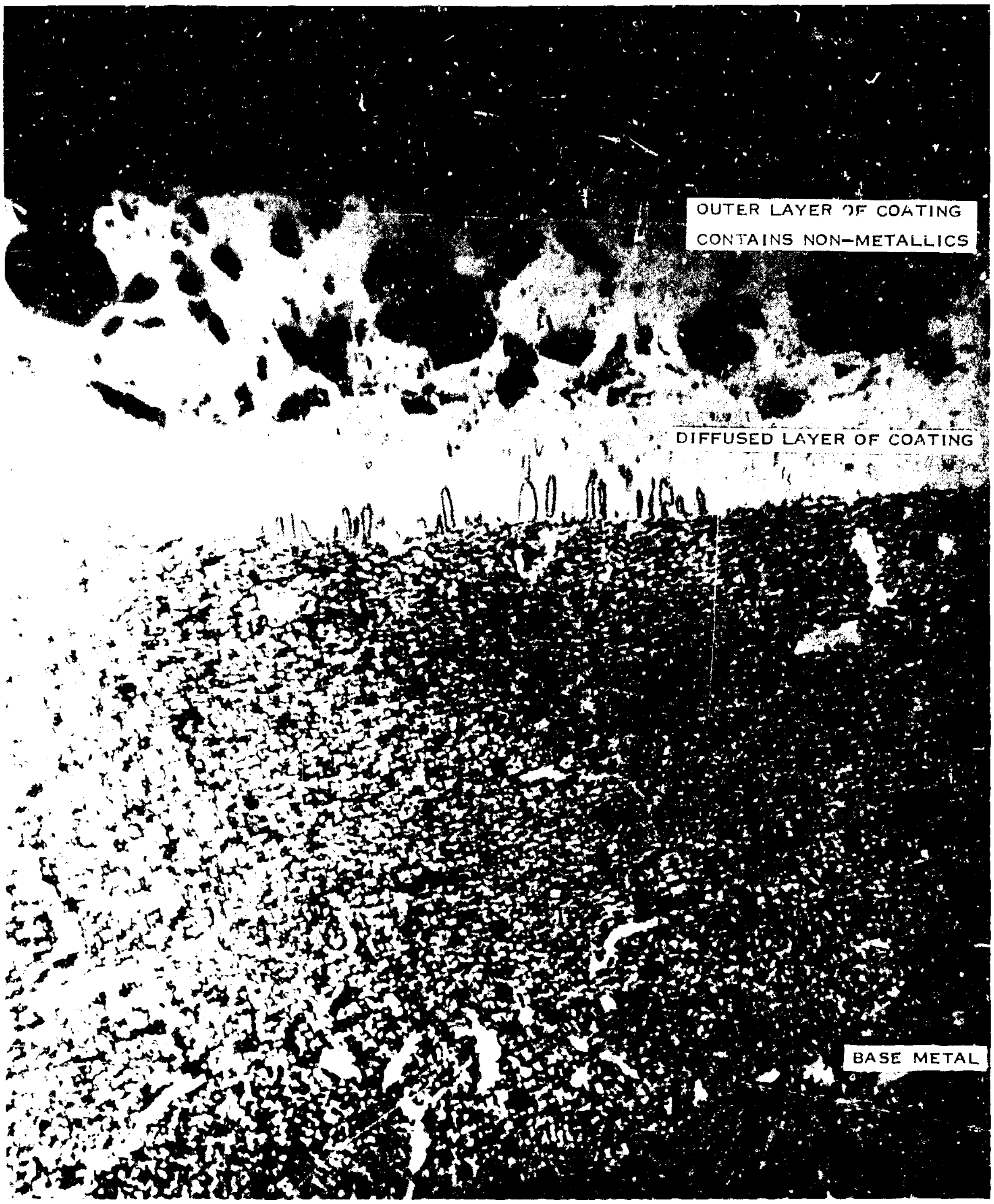


55 HOURS AT 2000 F TEST CONDITION WITH NO SEA SALT AND 0.4 WT % SULFUR
MARBLE'S REAGENT ETCH. 400X MAGNIFICATION

FIGURE 24
OXIDATION OF INCONEL 713C SPECIMEN

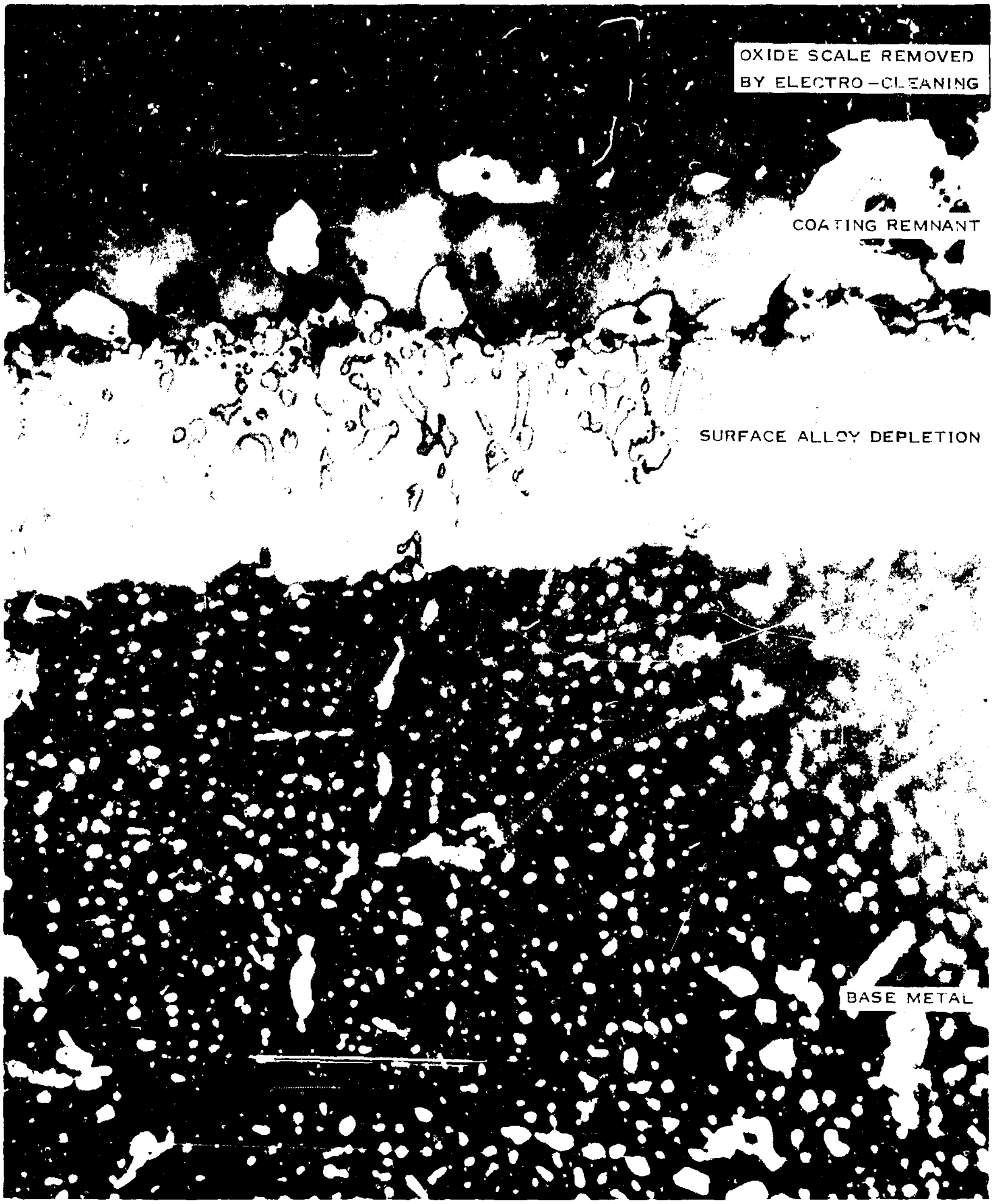
25 HOURS AT 2000 F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.4 WT % SULFUR
MARBLE'S REAGENT ETCH, 4000 X MAGNIFICATION

FIGURE 25
HOT CORROSION OF INCONEL 713C SPECIMEN



3% SULFURIC ACID—ELECTROLYTIC ETCH, 1000X MAGNIFICATION

FIGURE 26
UNEXPOSED MISCO MDC-1 COATED INCONEL 713C SPECIMEN



20 HOURS AT 2000 F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.040 WT % SULFUR
2% CHROMIC ACID-ELECTROLYTIC ETCH, 1000X MAGNIFICATION

FIGURE 27
DEPLETION OF MISCO MDC-1 COATED INCONEL 713C SPECIMEN

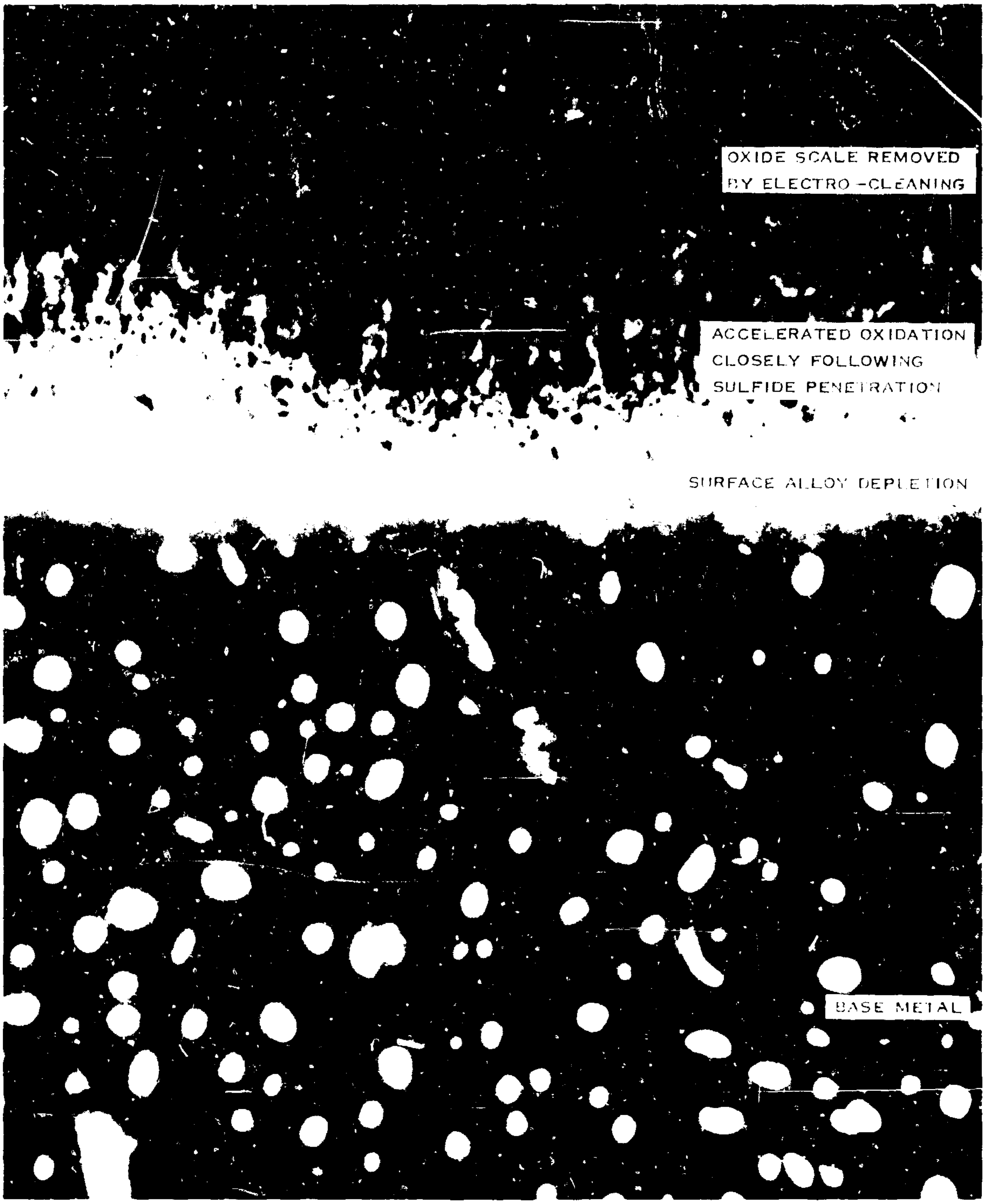
Once the MDC-1 coating was penetrated, the mode and intensity of attack observed was similar to that experienced by the bare Inconel 713C at comparable conditions of exposure. Characteristic micro-features of hot corrosion were found when the coating was penetrated by prolonged exposure in the presence of "sea salt", as shown in Figure 28.

The surface and matrix of a typical MDC-9 coated Inconel 713C specimen, in the as-received condition from Misco and as placed in test, is shown in Figure 29, The aluminum-chromium-type coating was applied to obtain a total depth of approximately 2 mils; which is divided about equally between an outer-layer and a diffusion-layer. As discussed in Appendix 2, the electro-cleaning procedure was found to alter the appearance of this coating, and so was not used to remove the surface scale of corrosion products from the test specimens following exposure during this investigation.

Test specimens of Inconel 713C were inspected by Misco using fluorescent penetrant (Zyglo) and certified to be free from cracks, as noted in Appendix 3. Similar inspection by Phillips following application of the MDC-9 coating by Misco revealed no cracks. However, it is of interest to note that a few cracks were found in the coating of a new, unexposed, specimen when sectioned for metallographic study. One such crack is shown in Figure 29, and it is pertinent to point out that characteristically the crack does not extend into the base alloy. It is speculated that these cracks were developed in the coating when the specimens were sectioned for the metallographic examination, and do not represent the as-received specimen condition. Indications are that the MDC-9 coating as applied by Misco to the Inconel 713C specimens used in this program was subject to cracking under stress.

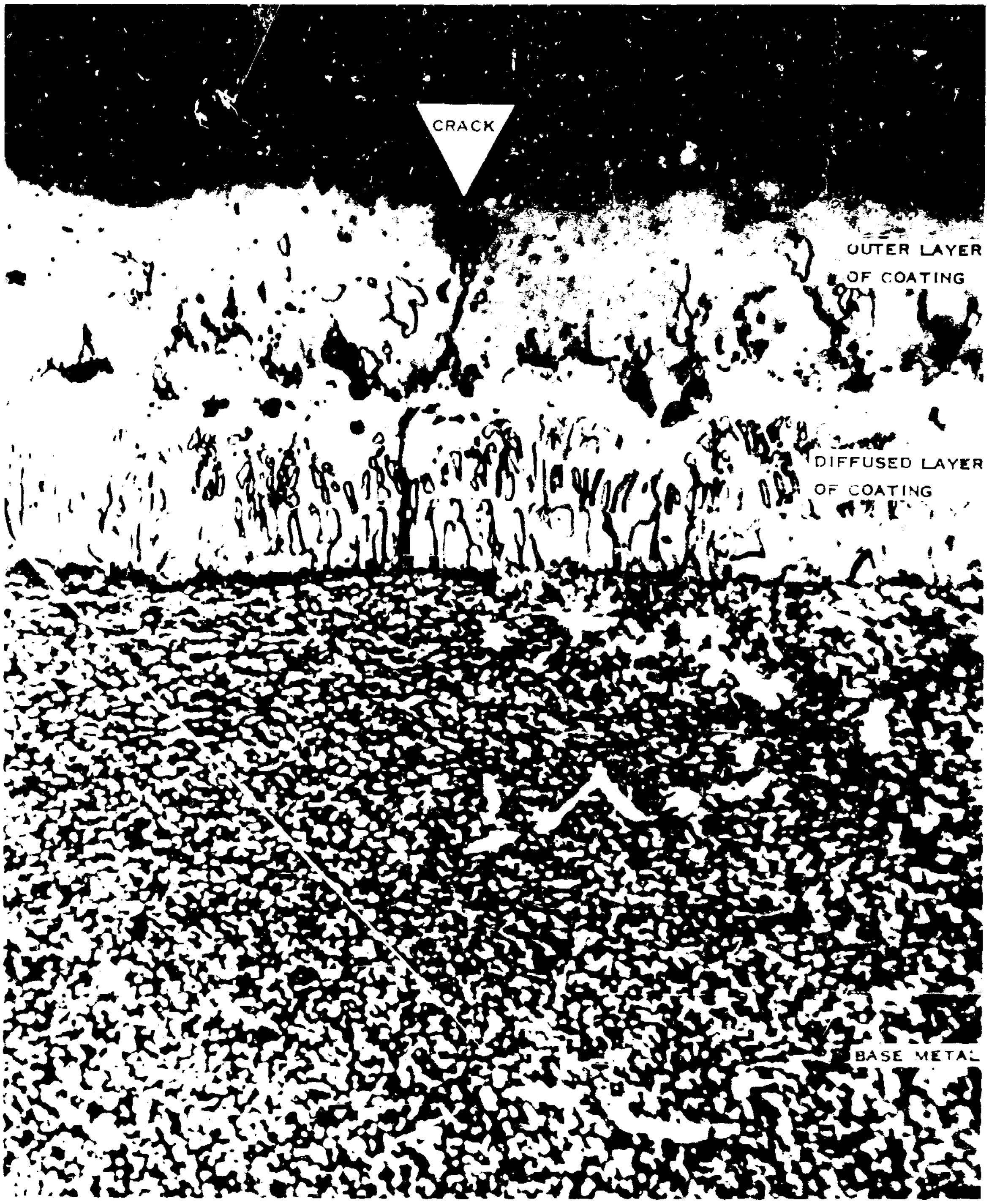
In those areas where the MDC-9 coating remained intact following exposure, evidence was found of alloy depletion in the diffused layer, as shown in Figure 30. This modification of the coating remnant is comparable to that experienced with the Misco MDC-1 coated Inconel 713C specimens, as previously described. Similarly, it was concluded that once the MDC-9 coating was penetrated, the corrosive attack was like that experienced by the bare Inconel 713C at comparable conditions of exposure.

In those areas where the MDC-9 coating was penetrated by hot corrosion the mode and intensity of attack was similar to that observed with bare Inconel 713C, as already described. Thus, it was suprising to find little or no evidence of sulfide penetration associated with the cracks which were found in the Misco MDC-9 coated Inconel 713C specimens following exposure. While there was evidence of alloy depletion around the cracks, as shown in Figure 31, it was not accompanied by the accelerated oxidation associated with hot corrosion. This indicates that the cracks do not serve as a focal point for the attack by corrosive materials over the range in conditions of exposure investigated.



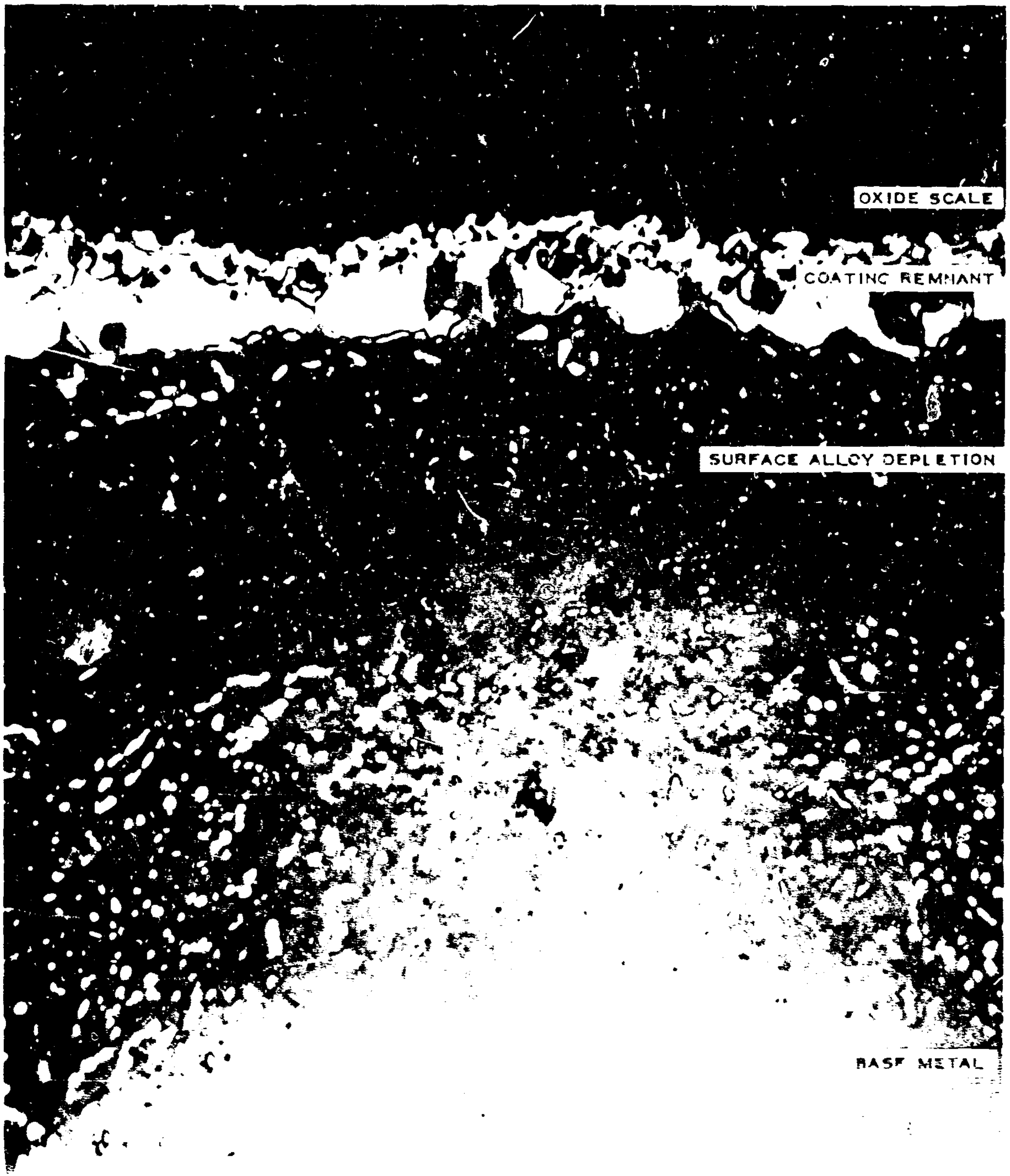
20 HOURS AT 2000 F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.040 WT % SULFUR
2% CHROMIC ACID–ELECTROLYTIC ETCH, 3000X MAGNIFICATION

FIGURE 28
HOT CORROSION OF MISCO MDC-1 COATED 713C SPECIMEN



3% SULFURIC ACID--ELECTROLYTIC ETCH, 1000X MAGNIFICATION

FIGURE 29
UNEXPOSED MISCO MDC-9 COATED INCONEL 713C SPECIMEN



25 HOURS AT 2000 F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.040 WT % SULFUR
2% CHROMIC ACID-ELECTROLYTIC ETCH, 400X MAGNIFICATION

FIGURE 30
DEPLETION OF MISCO MDC-9 COATED INCONEL 713C SPECIMEN

25 HOURS AT 2000 F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.040 WT % SULFUR.
3% SULFURIC ACID-ELECTROLYTIC ETCH. 400X MAGNIFICATION

FIGURE 31
CRACK IN MISCO MDC-9 COATED INCONEL 713C SPECIMEN

In general, the corrosive attack experienced by the superalloy and coatings during this investigation resulted in only shallow penetration of corrosion products, despite the catastrophic rates of metal loss encountered in the presence of "sea salt". The depth of surface penetration was usually less than one mil. This serves to justify the use of the metal-loss data as a valid measurement of the extent of corrosive attack on the superalloy and coatings over the range of conditions investigated.

5. FUTURE WORK

The effect of sulfur in fuel was found to vary among the various superalloys used in the previous programs (1); i.e., a reduction in fuel sulfur had no effect with some superalloys, and increased hot corrosion with two superalloys under some conditions. Inconel 713C, the base superalloy used for our coating study so far, was not affected significantly by changes in fuel sulfur content, while hot corrosion of SM-200 increased under some conditions with a reduction in fuel sulfur. A program will be conducted using Misco MDC-1 coated SM-200 to determine the effect of sulfur in fuel on the hot corrosion of a coating applied to a different base superalloy.

In previous programs (1) it was found that the effect of sulfur in fuel on hot corrosion varied with temperature. Statistically significant decreases in hot corrosion were found with reductions in fuel sulfur at temperatures below the melting point of sodium sulfate (1623 F) with superalloys that were unaffected by changes in sulfur content at higher temperatures. A program will be conducted to determine the effect of sulfur in fuel on the hot corrosion of one or more coated superalloys at temperatures below the melting point of sodium sulfate.

6. ACKNOWLEDGEMENTS

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Statistical Analysis by M. R. Goss and Lynn Jones.

Metallographic Analysis by E. H. Borgman and Velma Gooch.

7. REFERENCES

1. Schirmer, R. M. and Quigg, H. T., "Effect of JP-5 Sulfur Content on Hot Corrosion of Superalloys in Marine Environment". Progress Report No. 3 for Naval Air Systems Command Contract NOw 65-0310-d. Phillips Petroleum Company Research Division Report 4370-66R. Bartlesville, Oklahoma, July 1966, 173 pp.

2. Fromm, E. H., Quigg, H. T. and Schirmer, R. M., "Effect of JP-5 Sulfur Content on Hot Corrosion of Superalloys in Marine Environment". Progress Report No. 2 for Bureau of Naval Weapons Contract NOw 65-0310-d. Phillips Petroleum Company Research Division Report 4319-66R. Bartlesville, Oklahoma, January 31, 1966, 62 pp.

3. Quigg, H. T., Schirmer, R. M., "Effect of Manganese and Lead In JP Fuel on Hot Corrosion of Superalloys in Marine Environment". Progress Report No. 4 for Naval Air Systems Command Contract NOw-65-0310-d. Phillips Petroleum Company Research Division Report 4411-66R. Bartlesville, Oklahoma, August 1966.

4. Whitfield, M. G. and Parzuchowski, R. S., "Sulfidation Resistant Coatings". Preprint of Paper No. 115 presented at the Sixty-Ninth Annual Meeting of ASTM in Atlantic City, N. J., June 27 - July 1, 1966.

5. Danek, G. J., "State-of-the-Art Survey on Hot Corrosion in Marine Gas-Turbine Engines". U. S. Navy Marine Engineering Laboratory Report 32/65. Annapolis, Md. March 1965, 33 pp.

6. Fromm, E. H., "Design and Calibration of the Improved Phillips Fuel Testing Facilities". Phillips Petroleum Company Research Division Report 3527-63R. Bartlesville, Oklahoma, July 2, 1963.

7. Shirley, H. T., "Effects of Sulphate-Chloride Mixtures in Fuel-Ash Corrosion of Steels and High-Nickel Alloys". Journal of the Iron and Steel Institute, Vol. 82, February 1956, pp. 144-153.

8. Blade, O. C., "Aviation Fuel". Petroleum Product Survey Nos. 4, 9, 14, 19, 24, 29 and 34. Bartlesville, Oklahoma, Petroleum Research Center, Bureau of Mines, U. S. Department of the Interior, 1958, 1959, 1960, 1961, 1962, 1963 and 1964.

9. Hall, N. A., "Mean Specific Heats for the Working Media of Gas Turbine Powerplants". SAE Quarterly Transactions, Vol. 1 No. 3, pp 490-497, July 1947.

Research Division Report 4706-67R

8. APPENDIX 1
(Test Data)

The total weight loss, weight loss per unit area, and the visual ratings for Misco MDC-1 coated Inconel 713C specimens at the three concentrations of sulfur in fuel, in the absence of "sea salt" in air, are shown in Table 10. Similar data with 1.0 ppm "sea salt" in air are shown in Table 11. In Table 11, where two tests are shown for each sulfur level, the first test was conducted with specimen positions fixed in the cascade, and the second test was conducted with the specimens rotated among the three stages of the cascade at five-hour intervals. The data for tests with 10.0 ppm "sea salt" in air and three concentrations of sulfur in fuel are shown in Table 12.

The weight-loss data for Misco MDC-9 coated Inconel 713C specimens at the three levels of sulfur in fuel, in the absence of "sea salt" in air, are shown in Table 13. Similar data with 1.0 ppm "sea salt" in air are shown in Table 14. Specimen weights after exposure and sonic-cleaning were adjusted by the addition of 1.00mg/cm^2 to eliminate negative weight losses, and these adjusted values were used in our analyses.

The weight-loss data for uncoated Inconel 713C specimens at the three concentrations of sulfur in fuel, in the absence of "sea salt" in air, are shown in Table 15. Similar data with 1.0 ppm "sea salt" in air are shown in Table 16.

TABLE 10

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-1 COATED INCONEL 713C
(Zero "Sea Salt" in Air)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss		Visual Rating (b)
			mg	mg/cm^2	
<0.0040	6	5	44.4	2.19	N
<0.0040	3	10	31.8	1.57	N
<0.0040	4	15	35.2	1.74	N
<0.0040	2	20	45.6	2.25	N
<0.0040	5	25	42.5	2.10	N
<0.0040	5A	30	40.4	1.99	N
<0.0040	2A	35	37.1	1.83	N
<0.0040	4A	40	50.2	2.48	N
<0.0040	3A	45	40.3	1.99	N
<0.0040	6A	50	51.6	2.55	VLS
<0.0040	1	55	60.4	2.98	VLS
0.040	6	5	41.5	2.05	N
0.040	3	10	37.7	1.86	N
0.040	4	15	39.7	1.96	N
0.040	2	20	46.1	2.27	N
0.040	5	25	42.8	2.11	N
0.040	5A	30	48.3	2.38	VLS
0.040	2A	35	45.8	2.26	N
0.040	4A	40	62.3	3.07	N
0.040	3A	45	42.1	2.08	N
0.040	6A	50	49.5	2.44	N
0.040	1	55	65.3	3.22	N
0.40	6	5	38.6	1.90	N
0.40	3	10	29.2	1.44	VLS
0.40	4	15	26.1	1.29	VLS
0.40	2	20	43.0	2.12	VLS
0.40	5	25	49.6	2.45	VLS
0.40	5A	30	51.9	2.56	N
0.40	2A	35	32.8	1.62	LS
0.40	4A	40	85.4	4.21	VLS
0.40	3A	45	107.2	5.29	LS
0.40	6A	50	79.7	3.93	VLS
0.40	1	55	206.5	10.19	MS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) See Table 2 for rating system.

TABLE 11

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-1 COATED INCONEL 713C
(1.0 ppm "Sea Salt" in Air)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss mg	Test Specimen Weight Loss mg/cm^2	Visual Rating (b)
< 0.0040	6	5	56.5	2.79	VLS
< 0.0040	3	10	41.2	2.03	VLS
< 0.0040	4	15	73.0	3.60	LE
< 0.0040	2	20	65.9	3.25	VLS
< 0.0040	5	25	214.3	10.57	LE-LS
< 0.0040	5A	30	512.8	25.30	MS
< 0.0040	2A	35	113.3	5.59	LE
< 0.0040	4A	40	993.4	49.01	HE
< 0.0040	3A	45	308.8	15.23	MS
< 0.0040	6A	50	693.9	34.23	MS
< 0.0040	1	55	1150.3	56.75	MS
< 0.0040	2	5	46.4	2.29	N
< 0.0040	3	10	46.3	2.28	VLS
< 0.0040	6	15	58.2	2.87	VLS
< 0.0040	1	20	60.4	2.98	VLS
< 0.0040	4	25	72.4	3.57	VLS
< 0.0040	4A	30	156.8	7.74	LS
< 0.0040	1A	35	117.9	5.82	ME
< 0.0040	6A	40	84.2	4.15	LS
< 0.0040	3A	45	104.4	5.15	LS-LE
< 0.0040	2A	50	259.0	12.78	LS-LE
< 0.0040	5	55	200.4	9.89	LS
0.040	6	5	43.2	2.13	VLS
0.040	3	10	56.1	2.77	VLS
0.040	4	15	118.2	5.83	LS
0.040	2	20	483.9	23.87	MS
0.040	5A	20	123.5	6.09	LE
0.040	5	25	894.8	44.14	MS
0.040	2A	25	333.3	16.44	MS
0.040	4A	30	1556.2	76.77	MS
0.040	3A	35	1185.6	58.49	MS
0.040	6A	40	1983.5	97.85	...
0.040	1	45	4596.9	226.78	HS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) See Table 2 for rating system.

TABLE 11 (Continued)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss mg	Test Specimen Weight Loss mg/cm^2	Visual Rating (b)
0.040	2	5	173.3	8.55	LS
0.040	1A	5	59.8	2.95	VLS
0.040	3	10	254.7	12.56	LS
0.040	6A	10	97.2	4.80	VLE
0.040	6	15	233.8	11.53	VLE
0.040	3A	15	76.2	3.76	VLE
0.040	1	20	355.1	17.52	LS-LE
0.040	2A	20	141.8	7.00	LE
0.040	4	25	1127.3	55.61	MS
0.040	5	25	565.4	27.89	HS
0.40	6	5	70.9	3.50	N
0.40	3	10	62.1	3.06	VLS
0.40	4	15	131.6	6.49	LS-VLE
0.40	5A	15	65.3	3.22	VLS
0.40	2	20	225.2	11.11	LE
0.40	2A	20	659.2	32.52	MS
0.40	5	25	809.8	39.95	MS
0.40	4A	25	1558.1	76.87	MS
0.40	3A	30	340.3	16.79	MS
0.40	6A	35	1682.9	83.02	MS
0.40	1	40	4965.2	244.95	HS
0.40	2	5	47.5	2.34	VLS
0.40	1A	5	58.2	2.87	VLS
0.40	3	10	60.5	2.98	VLS
0.40	6A	10	62.6	3.09	VLS
0.40	6	15	126.4	6.24	MS
0.40	3A	15	60.3	2.97	VLS
0.40	2A	20	144.6	7.13	LE
0.40	1	20	270.5	13.34	MS
0.40	5	25	457.4	22.56	MS
0.40	4	25	1584.2	78.15	MS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) See Table 2 for rating system.

TABLE 12

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-1 COATED INCONEL 713C

(10.0 ppm "Sea Salt" in Air)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss		Visual Rating (b)
			mg	mg/cm²	
< 0.0040	2	5	60.0	2.96	VLS
< 0.0040	3A	5	216.9	10.70	ME-LS
< 0.0040	3	10	217.8	10.74	ME
< 0.0040	2A	10	3092.0	152.54	HS
< 0.0040	5	15	4205.8	207.48	HS
< 0.0040	4	15	2002.1	98.77	HS
< 0.0040	1	15	2349.9	115.93	HS
< 0.0040	6	15	5688.3	280.62	HS
0.40	2	5	84.8	4.18	VLS
0.40	3A	5	53.0	2.62	VLS
0.40	3	10	83.3	4.11	LS-C
0.40	2A	10	96.2	4.75	LS-C
0.40	1	15	1022.1	50.42	HS-C
0.40	4	15	1110.6	54.79	HE-LS-C
0.40	5	15	3157.6	155.77	HS-C
0.40	6	15	2582.9	127.42	HE-LS
4.0	2	5	36.2	1.79	LS
4.0	6	9	156.4	7.72	ME-LS
4.0	5	9	59.0	2.91	LS
4.0	3	9	71.0	3.50	LS-LE
4.0	4	9	194.9	9.62	ME-LS
4.0	1	9	1496.4	73.82	HE-LS
4.0	2	5	36.7	1.81	VLS
4.0	2A	8	63.2	3.12	VLS
4.0	3	10	87.2	4.30	LS-LE
4.0	5	13	101.1	4.99	LS
4.0	6	13	337.6	16.65	ME
4.0	1	13	258.4	12.75	LS
4.0	4	13	189.4	9.34	LS-VLE

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) See Table 2 for rating system.

TABLE 13

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR

MISCO MDC-9 COATED INCONEL 713C

(Zero "Sea Salt" in Air)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss mg	mg/cm^2	Adjusted (b)	Visual Rating(c)
< 0.0040	2	5	- 1.8	-0.09	0.91	N
< 0.0040	3	10	- 1.7	-0.08	0.92	N
< 0.0040	6	15	8.5	0.42	1.42	N
< 0.0040	1	20	5.7	0.28	1.28	N-C
< 0.0040	4	25	24.5	1.21	2.21	N-C
< 0.0040	4A	30	17.9	0.88	1.88	N
< 0.0040	1A	35	34.2	1.69	2.69	N-C
< 0.0040	6A	40	38.2	1.88	2.88	N-C
< 0.0040	3A	45	38.8	1.91	2.91	VLS-C
< 0.0040	2A	50	66.7	3.29	4.29	VLE-C
< 0.0040	5	55	22.3	1.10	2.10	N-C
0.040	2	5	- 1.8	-0.09	0.91	N
0.040	3	10	4.8	0.24	1.24	N
0.040	6	15	22.6	1.11	2.11	N
0.040	1	20	13.9	0.69	1.69	N
0.040	4	25	8.7	0.43	1.43	N-C
0.040	4A	30	29.1	1.44	2.44	N-C
0.040	1A	35	44.7	2.20	3.20	N-C
0.040	6A	40	30.0	1.48	2.48	N-C
0.040	3A	45	71.7	3.54	4.54	LS-C
0.040	2A	50	68.9	3.40	4.40	VLE-C
0.040	5	55	88.8	4.38	5.38	LS-C
0.40	2	5	-11.1	-0.54	0.46	N
0.40	3	10	-13.4	-0.66	0.34	N-C
0.40	6	15	-12.7	-0.63	0.37	N
0.40	1	20	- 8.3	-0.41	0.59	N
0.40	4	25	- 0.5	-0.02	0.98	N
0.40	4A	30	27.4	1.35	2.35	VLS-C
0.40	1A	35	38.3	1.89	2.89	VLS-C
0.40	6A	40	19.8	0.98	1.98	N
0.40	3A	45	4.3	0.21	1.21	N
0.40	2A	50	13.2	0.65	1.65	N
0.40	5	55	42.7	2.11	3.11	VLS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Weight loss adjusted by adding 1.00 mg/cm^2.

(c) See Table 2 for rating system.

TABLE 14

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-9 COATED INCONEL 713C
(1.0 ppm "Sea Salt" in Air)

Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss mg	mg/cm²	Adjusted (b)	Visual Rating (c)
< 0.0040	2	5	-1.9	-0.09	0.91	N
< 0.0040	3	10	6.8	0.34	1.34	N-C
< 0.0040	6	15	35.5	1.75	2.75	VLS-C
< 0.0040	1	20	41.1	2.03	3.03	VLE
< 0.0040	4A	20	36.2	1.79	2.79	VLS-C
< 0.0040	4	25	11.0	0.54	1.54	N-C
< 0.0040	1A	25	15.0	0.74	1.74	N-C
< 0.0040	6A	30	51.6	2.55	3.55	VLE-C
< 0.0040	3A	35	116.1	5.73	6.73	MS-C
< 0.0040	2A	40	406.5	20.05	21.05	MS-C
< 0.0040	5	45	190.8	9.41	10.41	MS-ME-C
0.040	2	5	-5.7	-0.28	0.72	N-C
0.040	3	10	3.5	0.17	1.17	N
0.040	4A	10	11.8	0.58	1.58	N-C
0.040	6	15	29.4	1.45	2.45	LS
0.040	1A	15	50.1	2.47	3.47	VLS-C
0.040	1	20	50.2	2.48	3.48	VLE
0.040	6A	20	88.0	4.34	5.34	LS-C
0.040	4	25	185.2	9.14	10.14	LE-C
0.040	3A	25	170.0	8.39	9.39	ME-C
0.040	2A	30	774.8	38.22	39.22	HS-C
0.040	5	35	729.1	35.97	36.97	HS-C
0.40	2	5	-4.9	-0.24	0.76	...
0.40	3	10	5.0	0.25	1.25	...
0.40	6	15	41.4	2.04	3.04	...C
0.40	1	20	-7.3	-0.36	0.64	...C
0.40	4A	20	218.9	10.80	11.80	LE
0.40	4	25	57.1	2.82	3.82	VLE
0.40	1A	25	1327.7	65.50	66.50	HS
0.40	6A	30	1807.1	89.15	90.15	HS
0.40	3A	35	915.2	45.15	46.15	MS-C
0.40	2A	40	1336.6	65.94	66.94	HS-C
0.40	5	45	2324.2	114.66	115.66	HS-C

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Weight loss adjusted by adding 1.00 mg/cm².

(c) See Table 2 for rating system.

TABLE 15

WEIGHT LOSS DATA FOR INCONEL 713C
(Zero "Sea Salt" in Air)

Sulfur In Fuel, wt %	Test Specimen Number	Total Exposure Time, hours	Total Specimen Weight Loss mg	Total Specimen Weight Loss mg/cm²
< 0.0040	2	5	76.7	3.78
< 0.0040	3	10	180.1	8.88
< 0.0040	6	15	310.1	15.30
< 0.0040	1	20	372.0	18.35
< 0.0040	4	25	467.8	23.08
< 0.0040	4A	30	857.3	42.29
< 0.0040	1A	35	950.7	46.90
< 0.0040	6A	40	1847.2	91.13
< 0.0040	3A	45	878.8	43.35
< 0.0040	2A	50	1516.4	74.81
< 0.0040	5	55	1269.5	62.63
0.040	2	5	102.8	5.07
0.040	3	10	203.7	10.05
0.040	6	15	363.1	17.91
0.040	1	20	514.5	25.38
0.040	4	25	863.1	42.58
0.040	4A	25	502.9	24.81
0.040	1A	30	615.2	30.35
0.040	6A	35	428.2	21.12
0.040	3A	40	722.5	35.64
0.040	2A	45	1381.6	68.16
0.040	5	50	1370.5	67.61
0.40	2	5	114.6	5.65
0.40	3	10	278.9	13.76
0.40	6	15	31.47	15.52
0.40	1	20	500.8	24.71
0.40	4	25	626.7	30.92
0.40	4A	30	949.1	46.32
0.40	1A	35	639.5	31.55
0.40	6A	40	1397.1	68.92
0.40	3A	45	980.1	48.35
0.40	2A	50	1640.4	80.93
0.40	5	55	1655.4	81.67

TABLE 16

WEIGHT LOSS DATA FOR INCONEL 713C
(1.0 ppm "Sea Salt" in Air)

Sulfur In Fuel, wt %	Test Specimen Number	Total Exposure Time, hours	Total Specimen Weight Loss mg	mg/cm^2
< 0.0040	2	5	175.8	8.67
< 0.0040	3	10	438.5	21.63
< 0.0040	6	15	1156.1	57.03
< 0.0040	4A	15	1371.5	67.66
< 0.0040	1A	20	1458.8	71.97
< 0.0040	1	20	1467.6	72.40
< 0.0040	4	25	2045.9	100.93
< 0.0040	6A	25	2321.0	114.50
< 0.0040	3A	30	1893.3	93.40
< 0.0040	2A	35	2642.6	130.37
< 0.0040	5	40	2576.6	127.11
0.040	2	5	312.4	15.41
0.040	4A	5	317.1	15.64
0.040	3	10	963.3	47.52
0.040	1A	10	399.8	19.72
0.040	6	15	1741.4	85.91
0.040	6A	15	1433.6	70.72
0.040	1	20	2942.7	95.84
0.040	3A	20	2346.2	115.74
0.040	4	25	4910.6	242.25
0.040	2A	25	4470.5	220.54
0.040	5	30	4117.9	203.15
0.40	2	5	560.8	27.67
0.40	1A	5	580.4	28.63
0.40	3	10	1481.8	73.10
0.40	6A	10	2682.0	132.31
0.40	6	15	2977.4	146.88
0.40	3A	15	2730.3	134.69
0.40	1	20	3706.4	182.85
0.40	2A	20	4043.9	199.50
0.40	4	25	7428.8	366.48
0.40	5	25	6070.1	299.46
0.40	5	5	848.3	41.85
0.40	4	5	1340.7	66.14

9. APPENDIX 2
(Test Equipment)

9.1. Test Facility

Phillips research facility for testing jet fuel, pictured in part in Figure 32 has been described previously in detail by Fromm (6). Air is supplied by rotary Fuller compressors and filtered by a Sales Vape-Sorber. This air is preheated just before it enters the 2-inch combustor testway by a Thermal Research heat-exchanger. Both fuel and "sea water" are supplied by nitrogen pressurization of their respective tanks. A portion of the metering and automatic control equipment can be seen in Figure 33. Air flow rates up to 2.0 lb/sec, at inlet air pressures up to 15 atmospheres, and inlet air temperatures up to 1400 F are attainable.

9.2. Phillips 2-Inch Combustor

A scale diagram of the 2-inch combustor used in this study is shown in Figure 34. Design details of the combustor are presented in Table 17. Basically, it embodied the principal features of combustors used in modern aircraft-turbine engines. It was a straight-through can-type, combustor with fuel atomization by a single, simplex-type, nozzle. The combustor liner was fabricated from 2-inch, Schedule 40, Inconel pipe, with added internal deflector skirts for film cooling of surfaces exposed to the flame.

A scale diagram of the Phillips test rig used in this study of hot corrosion is shown in Figure 35. Its design permits easy access to the fuel nozzle, combustor liner, test specimens, etc. The combustor installation was disassembled, inspected, and reconditioned after every test period.

Four chromel-alumel thermocouples were mounted on equal area centers at the location indicated in Figure 35, for measurement of exhaust gas temperature. The thermocouples were housed in $\frac{1}{4}$-inch diameter Inconel sheaths for protection.

The "sea water" injection point was located in the quench zone of the combustor, as indicated in Figure 35, rather than upstream of the combustor or in the primary-combustion zone. This avoided a severe corrosion problem with the combustor liner, and also insured exposure of test specimens to the desired "sea salt" concentration. The "sea water" was divided into two metered portions and introduced through opposing jets to obtain uniform distribution of "sea water" in air by impingment of the jet streams.

The exhaust section was water jacketed to obtain the desired durability of operation with high-temperature gases.

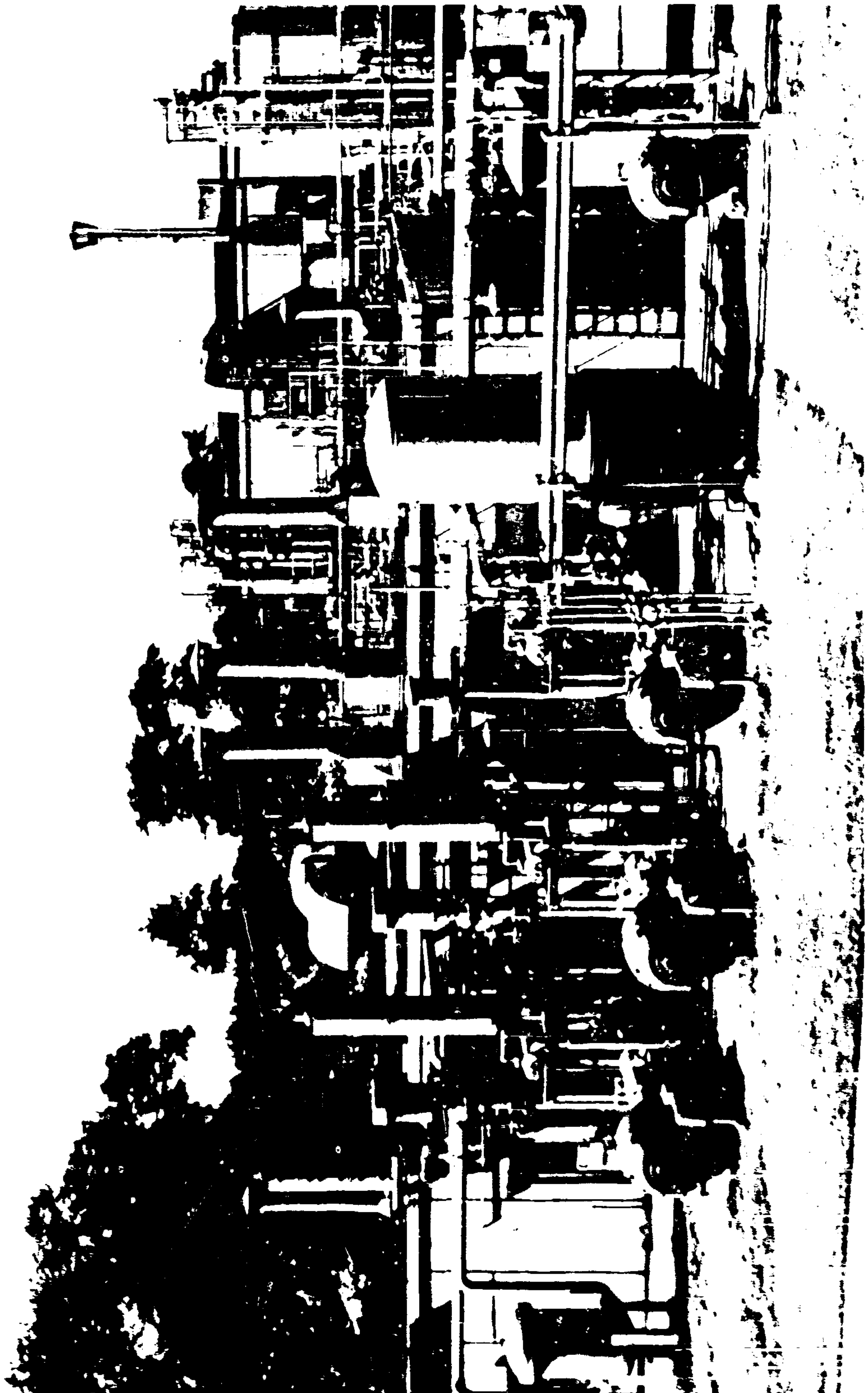


FIGURE 32
PHILLIPS RESEARCH FACILITY FOR JET FUELS

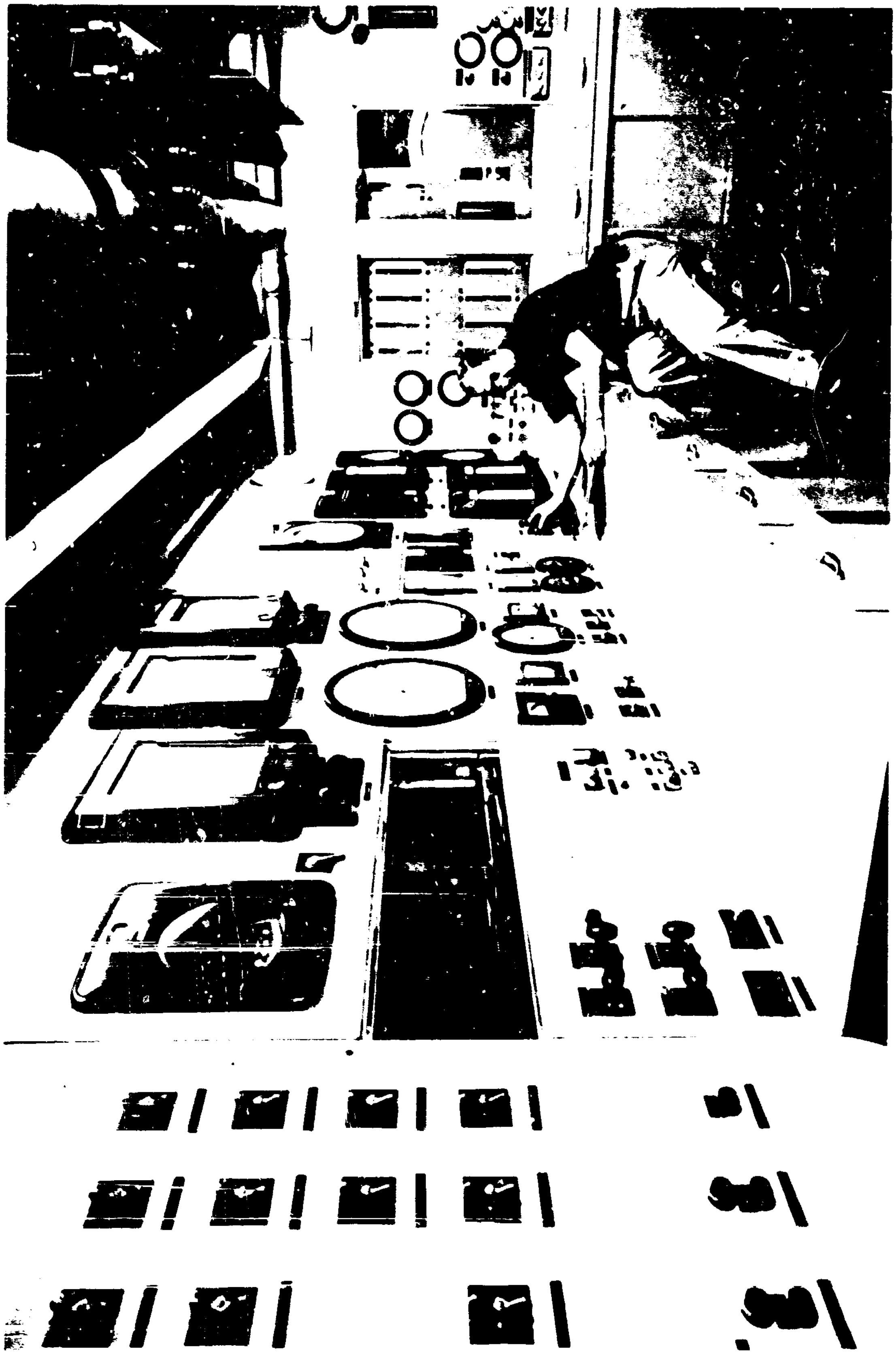


FIGURE 33
CONTROL ROOM FOR HIGH PRESSURE COMBUSTOR

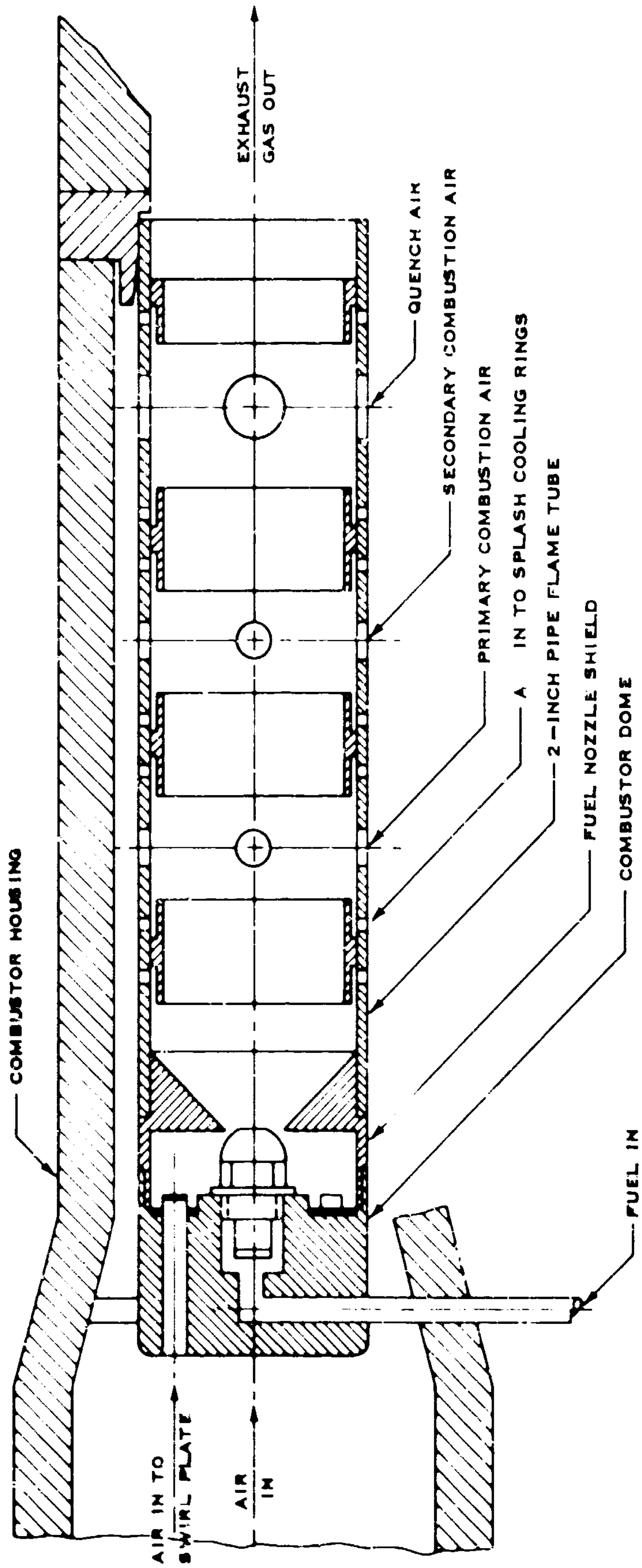


FIGURE 34
PHILLIPS 2 - INCH COMBUSTOR

TABLE 17

DESIGN DETAILS OF PHILLIPS 2-INCH COMBUSTOR

Combustor Configuration Number	15
Fuel Nozzle	
Type	Simplex (Monarch)
Spray Pattern	Semi-Solid Cone (PLP)
Spray Angle, degrees	50
Capacity, gph of No. 2 Fuel Oil @ 100 psi	13.8
Combustor Dome	
Air Inlet Type	Tangential Swirl
Shield Hole Diameter, in.	0.625
Total Hole Area, sq. in.	0.307
% Total Combustor Hole Area	8.7
Splash Cooling Air	
Hole Diameter, in.	0.125
Holes/Station	16
Number of Stations	7
Total Number of Holes	112
Total Hole Area, sq. in.	1.374
% Total Combustor Hole Area	38.7
Primary Combustion Air	
Hole Diameter, in.	0.250
Total Number of Holes	4
Total Hole Area, sq. in.	0.196
% Total Combustor Hole Area	5.5
Secondary Combustion Air	
Hole Diameter, in.	0.375
Total Number of Holes	4
Total Hole Area, sq. in.	0.442
% Total Combustor Hole Area	12.5
Quench Air	
Hole Diameter, in.	0.625
Total Number of Holes	4
Total Hole Area, sq. in.	1.227
% Total Combustor Hole Area	34.6
Total Combustor Hole Area, sq. in.	3.546
% Cross Sectional Area	133.4

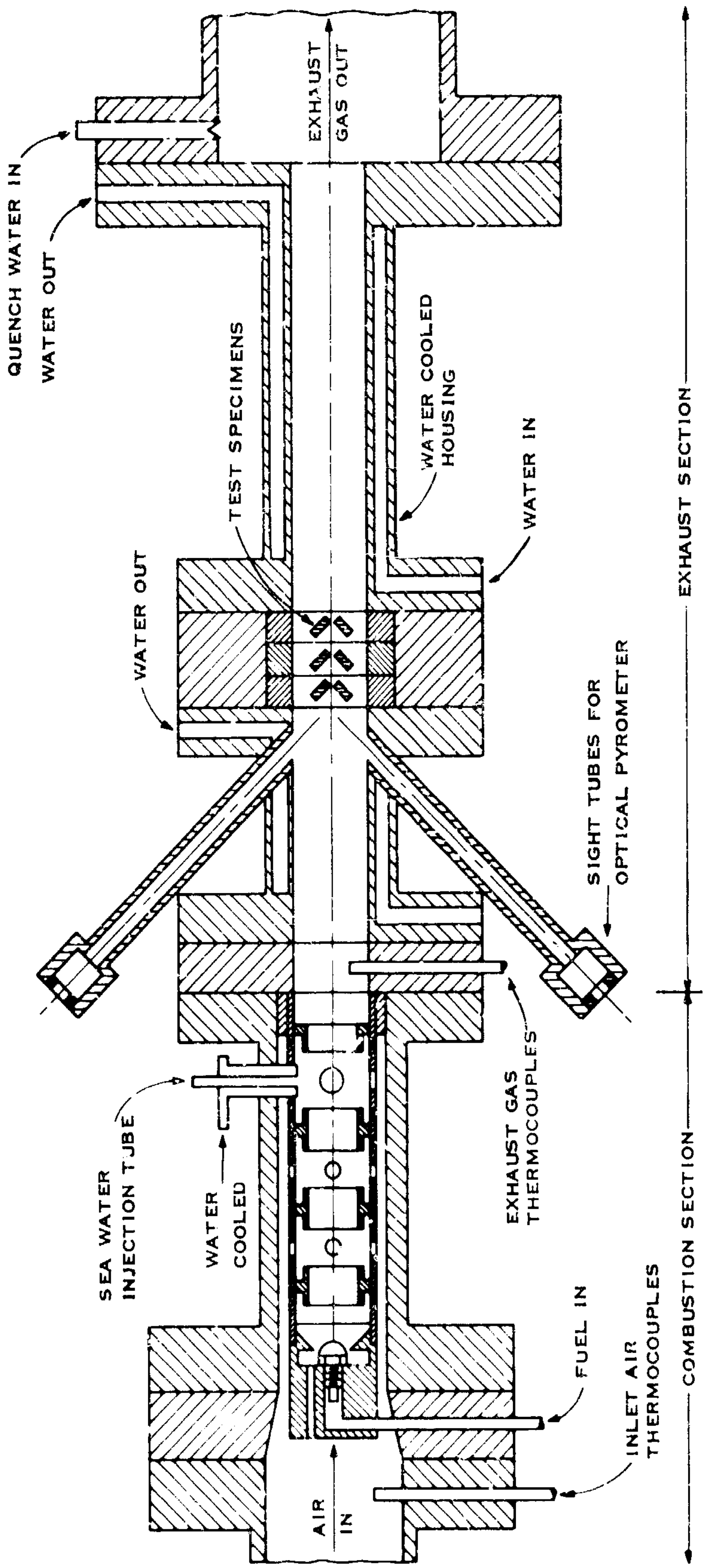


FIGURE 35
PHILLIPS TEST RIG FOR HOT CORROSION STUDIES

9.3. Specimen Holders

The general location of the specimen holders in the exhaust gas from the 2-inch combustor is shown in Figure 35. They are separated from the 2-inch combustor by a 6-inch, water-cooled, spool; and are followed by another 12-inch, water-cooled, spool prior to quench-water injection. Each holder accomodates two metal strips, 1/8 by 1/2 by 2-3/8 inches, as shown in Figure 36. Three holders were combined in a cascade, as shown in Figure 37, with each successive holder rotated 120 degrees to prevent channeling of the hot-gas flow. A picture of the specimens mounted in the cascade is shown in Figure 38.

The cross-sectional area of the 2-inch pipe in which the specimen holders are located is 3.36 square inches; however, the unblocked area in the specimen holder is only 1.59 square inches. The holders maintain the specimens at an angle of 45 degrees to the axis of the pipe in which they are located. This provides for acceleration of the gas flow over the surface of the specimens, much as over the turbine blading in an actual engine. The specimens are subjected to appreciable gas-pressure loading while the test is in progress. It is sufficient to slightly bow the Inconel 713C specimens at the 2000 F test condition, and the effect increases with exposure time.

The test specimens mounted in the first stage of the cascade were aligned with ½-inch I. D. sighting tubes as shown in Figure 35. This allowed for measurement, if desired, of their brightness temperature, using a Leeds and Northrup Model 8622-S optical pyrometer.

9.4. Specimen Cleaning

New specimens were cleaned by vapor degreasing with trichloroethylene, using the apparatus shown diagramatically in Figure 39. Subsequently the specimens were handled with degreased stainless-steel tongs. The initial weight of each test specimen was determined following degreasing.

After exposure, cleaning was necessary to remove the frequently heavy accumulation of surface deposit or scale, to allow for the measurement of metal loss by the specimens from hot corrosion.

In previous studies (1) an electro-cleaning technique was used which has been described by Shirley (7). Briefly, the electro-cleaning technique consists of immersing the specimens in molten sodium hydroxide at 750 to 790 F with 1/3 amp/sq cm passing through the specimen for a period of 10 minutes. The specimens were scrubbed with a wire brush during a water quench, rinsed in acetone, dried, and reweighed. The apparatus used for electro-cleaning is shown diagrammatically in Figure 40. Provisions were made to permit electro-cleaning, simultaneously, of six specimens. This technique was used for cleaning Misco MDC-1 coated Inconel 713C specimens and bare Inconel 713C specimens after exposure during the current programs.

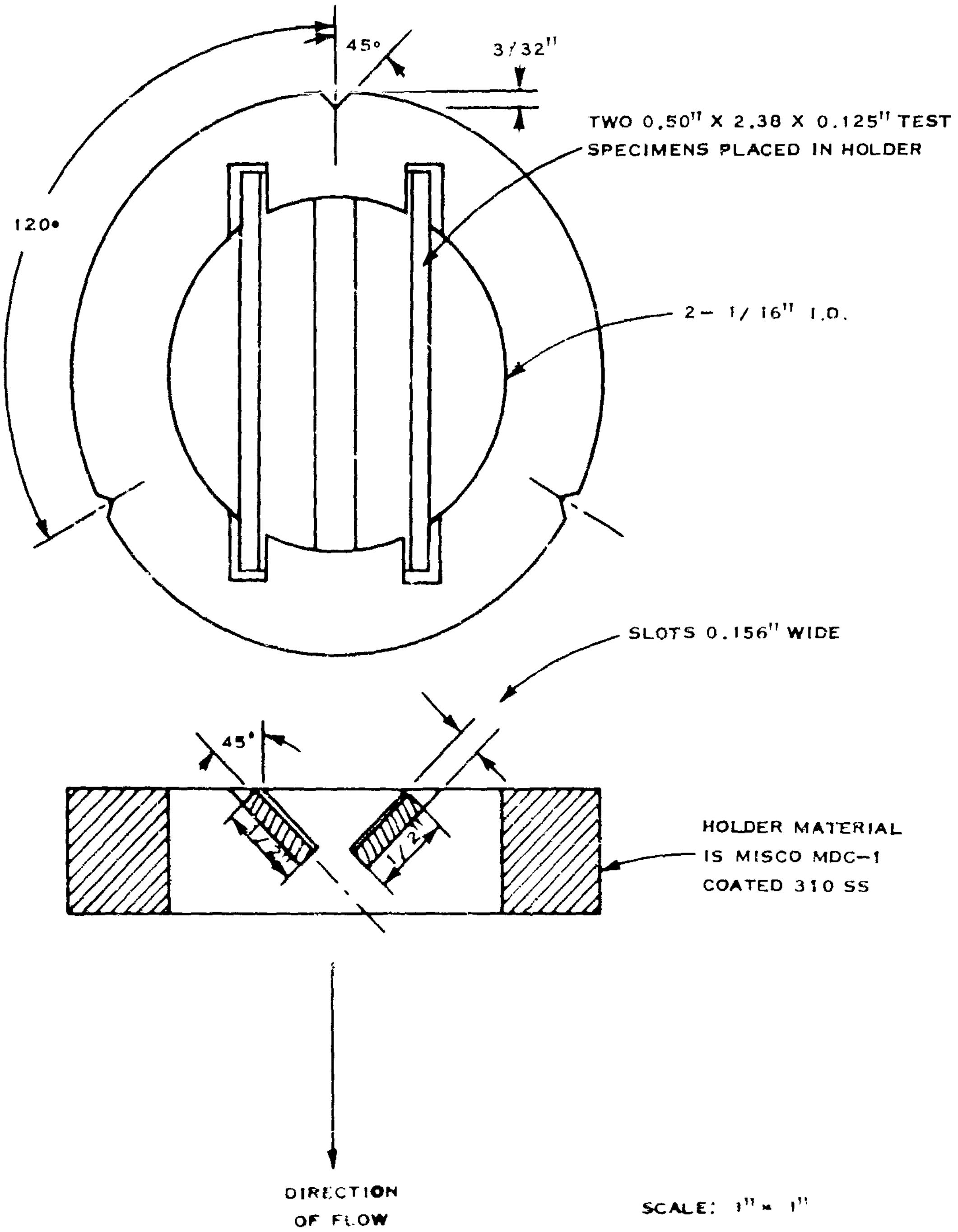


FIGURE 36
TEST SPECIMEN HOLDER

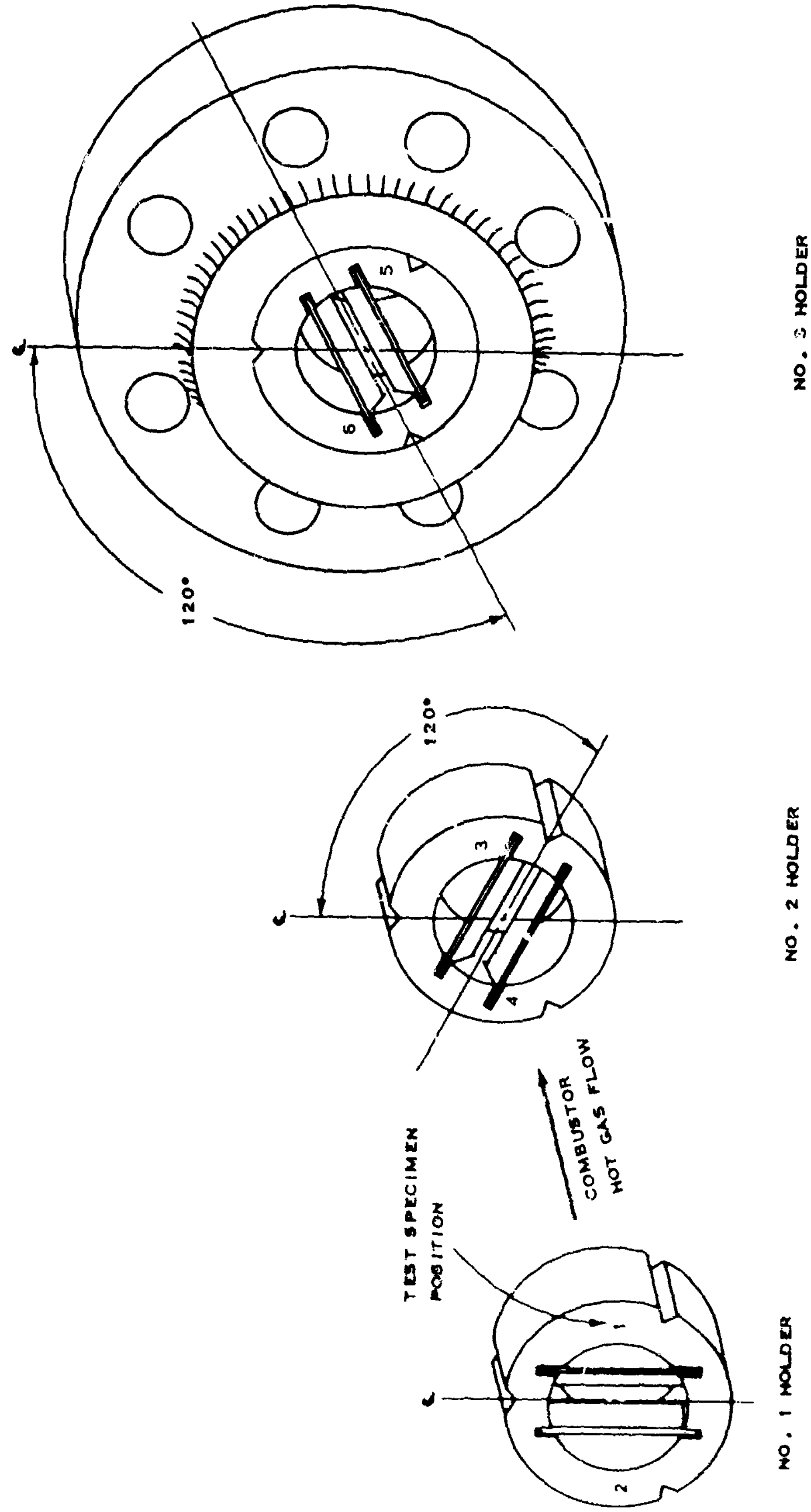


FIGURE 37
CASCADE ASSEMBLY FOR TEST SPECIMENS

2X MAGNIFICATION

FIGURE 38
SPECIMENS MOUNTED IN CASCADE

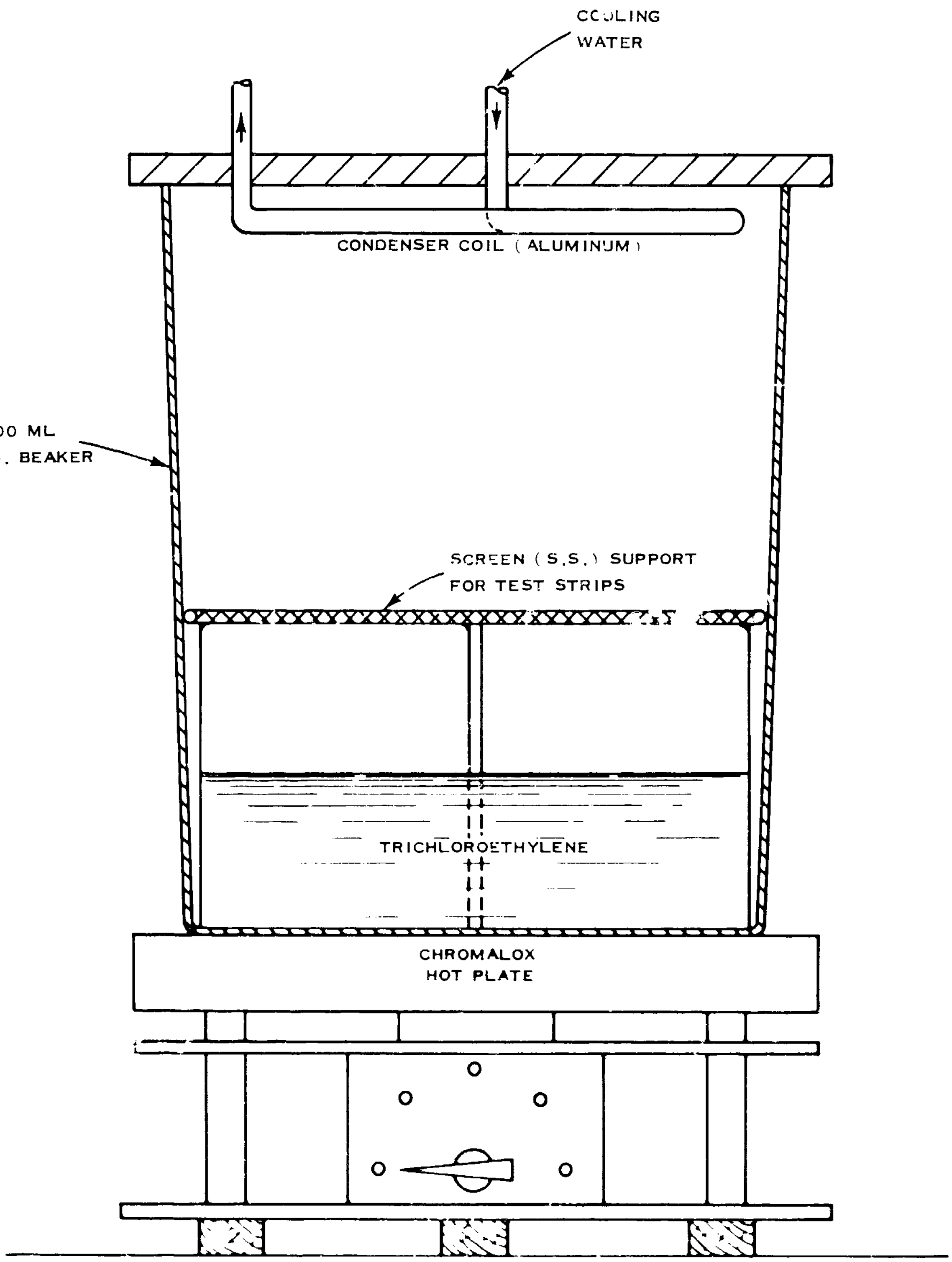


FIGURE 39
APPARATUS FOR VAPOR DEGREASING OF CORROSION
TEST SPECIMENS PRIOR TO TEST

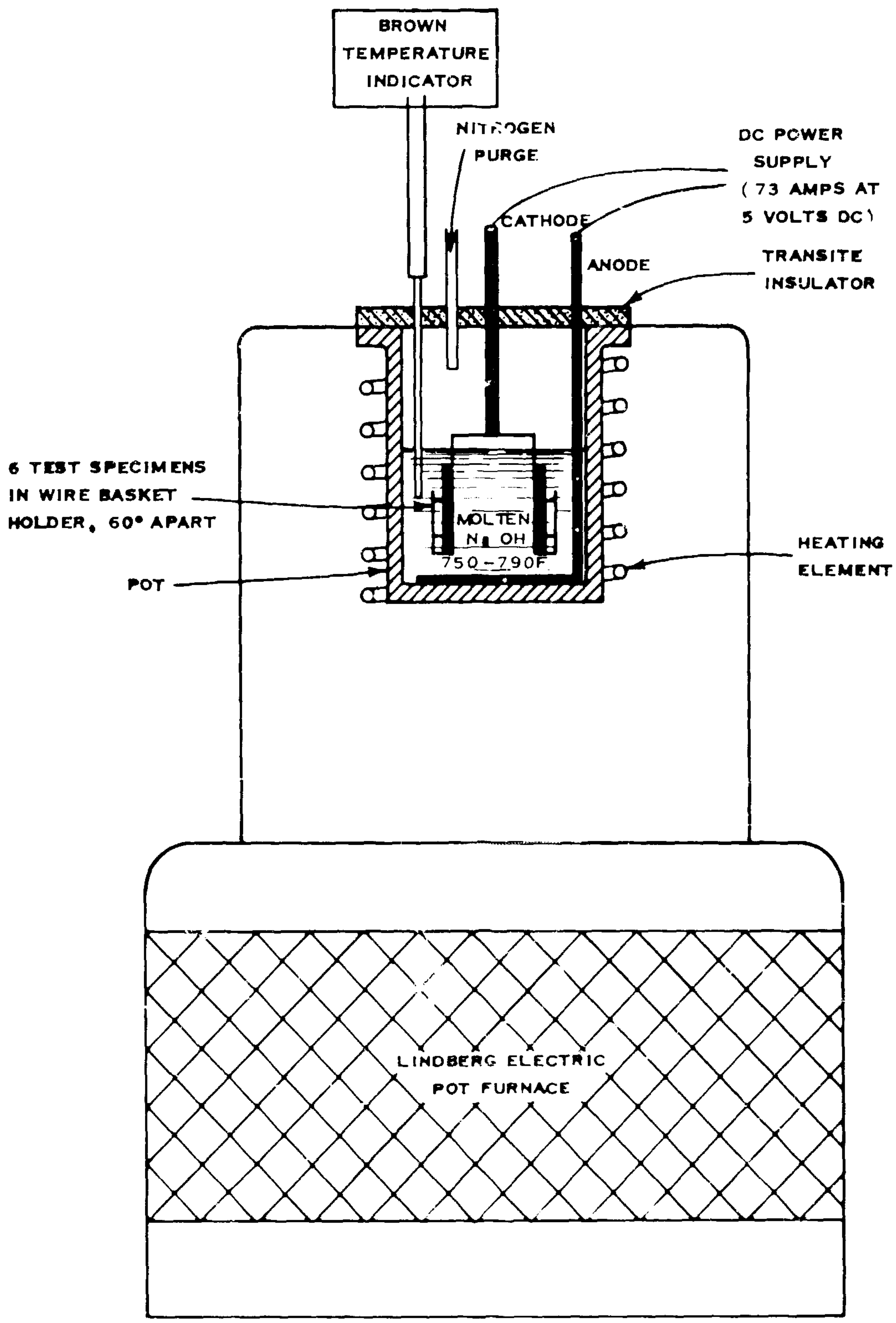


FIGURE 40
CATHODIC DESCALING APPARATUS FOR CLEANING
TEST SPECIMENS FOLLOWING EXPOSURE TO HOT GAS CORROSION

In previous studies (1) it was shown that loss of weight of unexposed test specimens after electro-cleaning was insignificant for uncoated specimens, but cleaning loss from unexposed Misco MDC-1 coated Inconel 713C specimens averaged 1.2 mg/cm^2. In the current program, extrapolation of the curves of weight loss with time to zero hours of exposure also indicats a weight loss of one mg/cm^2, or more. To eliminate the possible loss of material by electro-cleaning, it was decided to clean the Misco MDC-9 coated Inconel 713C specimens by water washing, using deionized water in an ultrasonic bath for 90 minutes. Specimens were then brushed with a fiber brush, rinsed in acetone, dried, and weighed. After cleaning the specimens from the first test condition, it was found that two of the specimens showed a slight gain in weight, indicating incomplete removal of corrosion scale or "sea salt" residue, or both, from the specimens. The specimens were then electro-cleaned and reweighed. The weight-loss data obtained with both water-washing and electro-cleaning, for each specimen from the first test, are shown in Table 18.

Visual inspection of the Misco MDC-9 coated Inconel 713C specimens revealed that most of the coating had been removed by electro-cleaning. Upon investigation, it was found that the temperature of the caustic bath used for cleaning had inadvertently been at an estimated 900 F, rather than at the standard 750 to 790 F. To further evaluate the effect of these cleaning techniques on loss of coating, a number of unexposed test specimens of bare Inconel 713C, Misco MDC-1 and MDC-9 coated Inconel 713C were cleaned and their weight losses are shown in Table 19. As expected, the water-wash in the ultrasonic bath did not remove a significant amount of material. The uncoated Inconel 713C was relatively insensitive to temperature of the caustic bath, and weight loss was low in all cases. However, both Misco MDC-1 and MDC-9 coatings were sensitive to caustic temperature, with the Misco MDC-9 coated Inconel 713C specimens being affected the most.

Upon metallographic examination, modification of the interface between the diffused layer of the coating and the base metal was observed to result from electro-cleaning Misco MDC-9 coated Inconel 713C specimens. This is illustrated by the photomicrographs shown in Figure 41, where a comparison can be made of the coating on a new test specimen, unexposed, (Figure 41A) with that on a new test specimen which had been electro-cleaned at 750 F (Figure 41B) and 900 F (Figure 41C). The nature of the phase changes observed has not been established.

A decision was made to use the water-wash (sonic-cleaning) technique for cleaning the Misco MDC-9 coated Inconel 713C specimens in this program. To permit an analysis such as used in previous programs, based on logarithms of weight loss, the weight-loss values were adjusted by the addition of 1.00 mg/cm^2 to all values to eliminate negative weight losses.

TABLE 18

EFFECT OF CLEANING TECHNIQUE ON WEIGHT LOSS

BY MISCO MDC-9 COATED INCONEL 713C

(0.40 wt % Sulfur in Fuel and 1.0 ppm Sea Salt in Air)

Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss: Water Washed (b) mg	mg/cm^2	Electro-Cleaned (c) mg	mg/cm^2	Difference (d) mg/cm^2
(e)	0	0.8	0.04	220.5	10.88	10.84
2	5	-4.9	-0.24	276.4	13.64	13.88
3	10	5.0	0.25	207.3	10.23	9.98
6	15	41.4	2.04	227.2	11.21	9.17
1	20	-7.3	-0.36	168.7	8.32	8.68
4A	20	218.9	10.80	373.3	18.42	7.62
4	25	57.1	2.82	170.5	8.41	5.59
1A	25	1327.7	65.50	1477.2	72.87	7.37
6A	30	1807.1	89.15	1966.1	96.99	7.84
3A	35	915.2	45.15	1108.5	54.69	9.54
2A	40	1336.6	65.94	1482.4	73.13	7.19
5	45	2324.2	114.66	2503.1	123.48	8.82

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Washed in deionized water in ultrasonic bath for 90 minutes.

(c) Caustic bath temperature believed to have been 900 F.

(d) Difference in weight loss between electro-cleaned and water washed techniques.

(e) New test specimen, unexposed.

TABLE 19

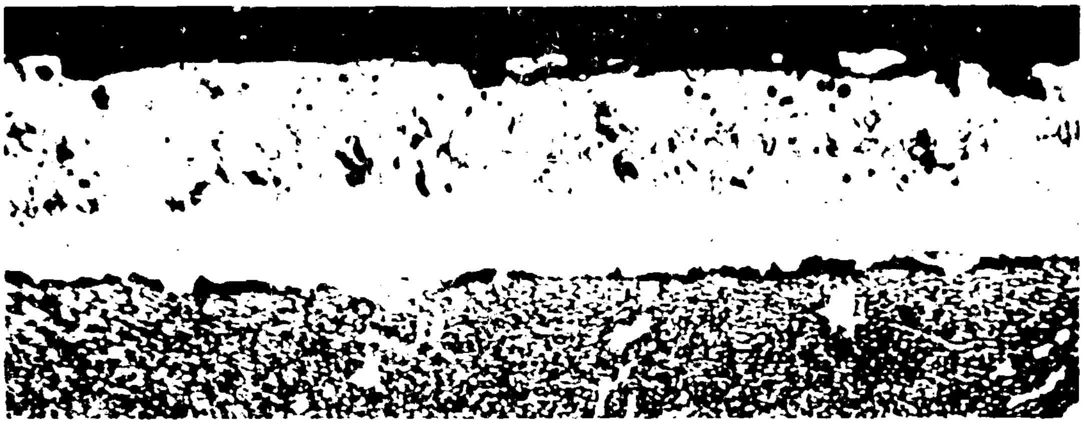
COMPARISON OF SPECIMEN CLEANING TECHNIQUES

		Test Specimen Weight Loss					
		WATER WASH		ELECTRO		TOTAL	
Superalloy	Coating	mg	mg/cm2	mg	mg/cm2	mg	mg/cm2
Inconel 713C	...	0.3	0.02	5.6(a)	0.28	5.9(a)	0.29
Inconel 713C	Misco MDC-1	1.2	0.06	58.5(a)	2.89	59.7(a)	2.94
Inconel 713C	Misco MDC-9	1.0	0.05	77.4(a)	3.82	78.4(a)	3.87
Inconel 713C	...	...	...	5.4(a)	0.27	5.4(a)	0.27
Inconel 713C	Misco MDC-1	...	...	64.6(a)	3.19	64.6(a)	3.19
Inconel 713C	Misco MDC-9	...	...	87.8(a)	4.33	87.8(a)	4.33
Inconel 713C	...	...	...	3.4(b)	0.17	3.4(b)	0.17
Inconel 713C	Misco MDC-1	...	...	28.4(b)	1.40	28.4(b)	1.40
Inconel 713C	Misco MDC-9	...	...	0.3(b)	0.51	10.3(b)	0.51

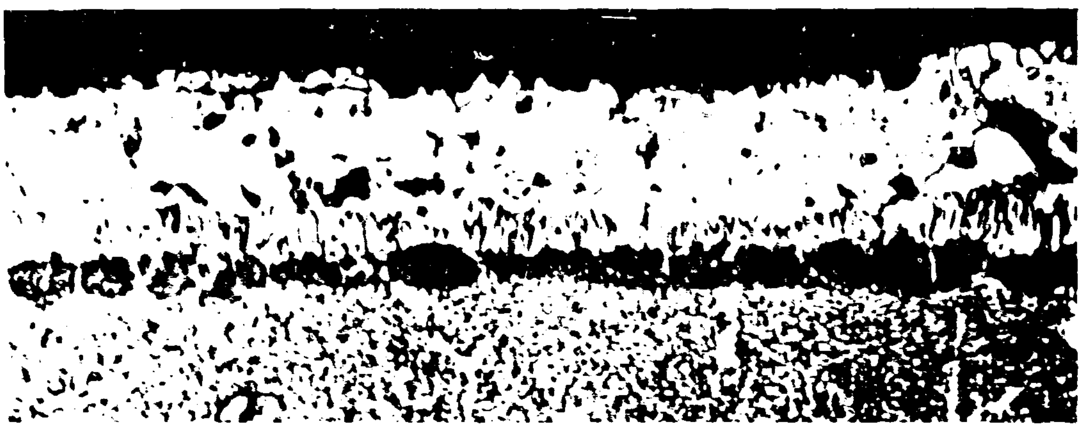
(a) Cleaning bath temperature 900 F

(b) Cleaning bath temperature 750 F

A. NEW TEST SPECIMEN UNEXPOSED



B. NEW TEST SPECIMEN ELECTRO-CLEANED AT [illegible]50 F



C. NEW TEST SPECIMEN ELECTRO-CLEANED AT [illegible] F

3% [illegible] ACID-ELECTROLYTE [illegible] MAGNIFICATION

FIGURE 41
MODIFICATION OF MISCO MDC-9 COATING
ON INCONEL 713C BY ELECTRO CLEANING

10. APPENDIX 3
(Test Materials)

10.1. Test Fuels

10.1.1. Sulfur in JP-5

The sulfur content of 197 samples of grade JP-5 aviation turbine fuel, representative of domestic and foreign purchases by the United States Navy from September 1966 to February 1967 had a median value of 0.04 weight per cent. The cumulative distribustions for West Coast, East and Gulf Coasts, Foreign, and Total are shown in Figure 42. These data indicate that a substantial, order-of-magnitude, reduction in the sulfur limit for JP-5 fuel could drastically curtail availability, unless accompanied by incentives for modernization of manufacturing techniques.

In previous investigations (1) three fuels were used having successive, order-of-magnitude, reductions in sulfur concentration, starting at the JP-5 sulfur limit; i.e., 0.40, 0.040, and <0.0040 weight per cent sulfur. These three levels of sulfur concentration span the range for samples in the survey, with the intermediate level of 0.040 weight per cent at the median value. It was decided that the current investigation would be conducted using the same three sulfur concentrations in fuel.

10.1.2. Base Fuel

The base fuels selected for use in this investigation were segregated samples of production ASTM Type A aviation-turbine fuel. The physical and chemical properties of interest to this investigation are presented in Table 20. The average values of pertinent properties from the Bureau of Mines Product Survey (7) over the period from 1957 through 1964 are also shown for grade JP-5 aviation turbine fuel. The physical and chemical properties of the base fuel closely approximate the average for JP-5, with the exception of its very low sulfur content. The base fuel also was analyzed for metal content, to be certain that its iron, vanadium, nickel, and copper contents were negligible; if present, they would concentrate as ash and might alter the scale composition on the test specimens exposed to the exhaust gases.

The base fuels are essentially free of sulfur, containing less than 0.0040 weight per cent. Two fuels of higher sulfur contents were produced by blending to 0.040 and 0.40 per cent by weight of sulfur using ditertiary butyl disulfide.

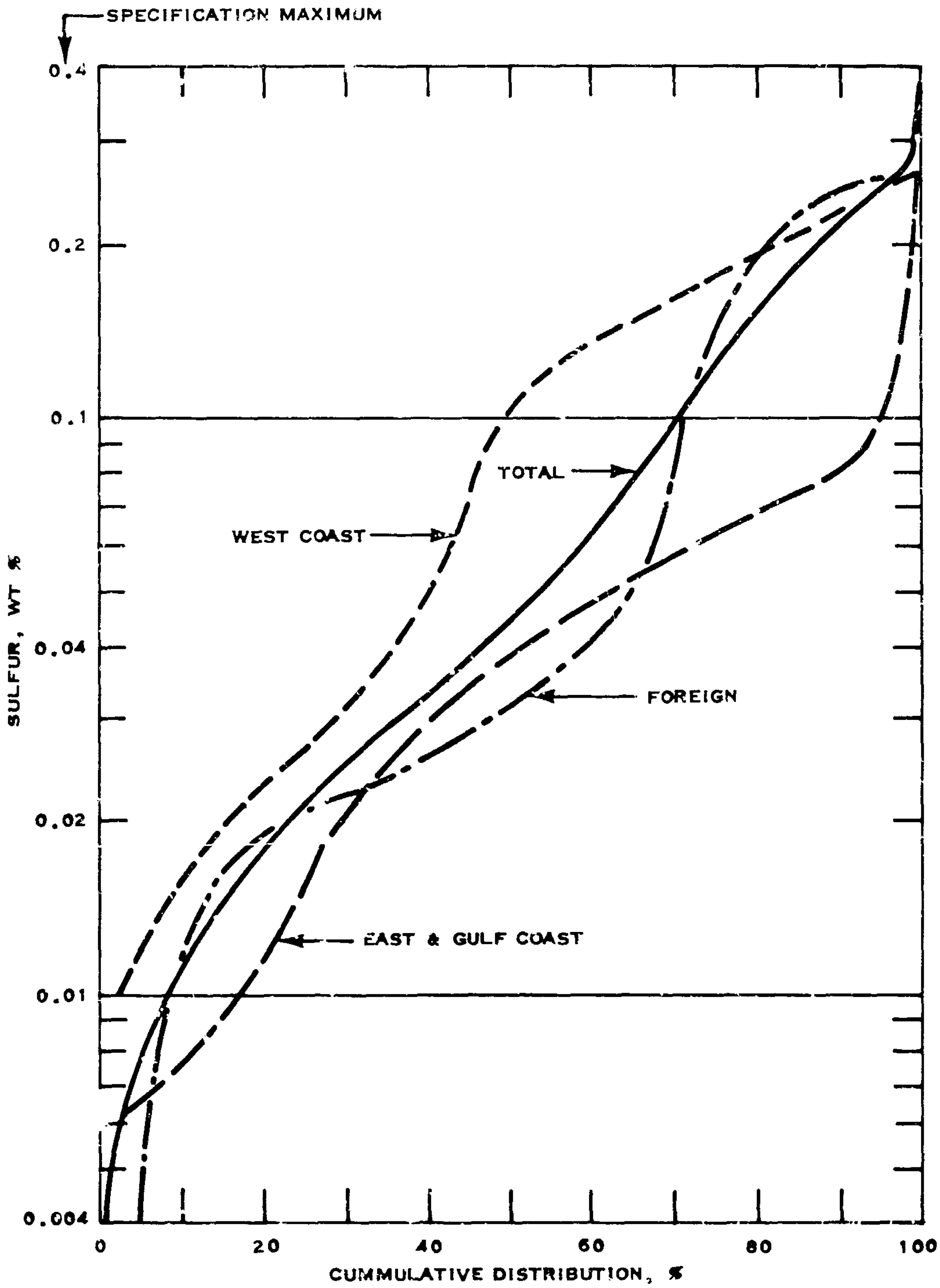


FIGURE 42
SULFUR CONTENT OF JP-5 FUEL PURCHASES
(SEPTEMBER 1966 – FEBRUARY 1967)

TABLE 20

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

	Test Fuel Base (a)	Test Fuel Base (b)	Average JP-5 (c)
Distillation Temperature, F			
Initial Boiling Point	332	326	
5 volume per cent evaporated	350	352	
10 volume per cent evaporated	355	357	
20 volume per cent evaporated	366	358	382
30 volume per cent evaporated	376	377	
40 volume per cent evaporated	388	388	
50 volume per cent evaporated	399	398	413
60 volume per cent evaporated	412	410	
70 volume per cent evaporated	427	423	
80 volume per cent evaporated	443	438	
90 volume per cent evaporated	462	457	455
95 volume per cent evaporated	476	473	
End Point	496	493	
Gravity, degrees API	45.8	45.9	42.1
Gum, milligrams per 100 mls.	0.6	0.5	1.0
Smoke Point, millimeters	27.0	26.2	23.1
Hydrogen Content, weight per cent	13.8	14.0	13.6
Composition, weight per cent			
Sulfur	< 0.0040(d)	< 0.0040(d)	0.10
Metals (e)			
Iron	< 0.0001	< 0.0001	
Vanadium	< 0.0001	< 0.0002	
Nickel	< 0.0001	< 0.0001	
Copper	6×10^{-7}	38×10^{-7}	
Hydrocarbon Types			
Normal Paraffins	27 (f)	27 (f)	
Isoparaffins	23 (f)	23 (f)	
Cycloparaffins	36 (f)	36 (f)	
Olefins	0.40	0.33	
Aromatics	15.80	13.37	14.3

Notes:

(a) Segregated sample (BJ65-8-G8) of production ASTM Type A aviation turbine fuel, processed from West Texas crude and finished by hydrotreating.
(b) Segregated sample (BJ66-8-G7) of production ASTM Type A aviation turbine fuel, processed from West Texas crude and finished by hydrotreating.
(c) U.S. Bureau of Mines Petroleum Product Survey (Ref. 8).
(d) Higher sulfur content test fuel obtained by blending to desired sulfur level using ditertiary butyl disulfide.
(e) X-ray fluorescence analysis.
(f) Typical value for this product.

10.2. "Sea Water"

10.2.1. Composition

A synthetic "sea water" was used in this study. Its formulation was taken from the Standard Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water, ASTM Designation D-665-60. The components and their concentrations are shown in Table 21. "Sea water" and "sea salt" (i.e., in quotes) are used throughout this report to indicate the synthetic composition shown in Table 21.

As discussed in Reference 1, the abundance of various elements in the synthetic formula compares very favorably with the average sea water composition. The one exception is silicon, and its exclusion from the synthetic "sea water" seems justified in the light of its reported variation in abundance from one water-mass to another by a factor of 1000, or more.

10.2.2. Ingestion Rate

As discussed in Reference 1, establishing a realistic level for the concentration of "sea salt" in the air ingested by a gas-turbine engine operating in a marine environment is difficult from available literature. However, it was concluded that an ingestion rate of 1.0 ppm "sea salt" in air was a realistic level. During the first two quarters under this contract studies were conducted on the effect of sulfur in fuel on hot corrosion of Misco MDC-1 and Misco MDC-9 coatings on Inconel 713C using two of the three levels of "sea salt" in air used in previous investigations (zero and 1.0 ppm "sea salt" in air). These two concentrations of "sea salt" in air were selected for the current evaluation using uncoated Inconel 713C.

Exploratory tests reported in Reference 2 were conducted with two levels of sulfur in fuel and 10.0 ppm "sea salt" in air to evaluate the effect on hot corrosion of Misco MDC-1 coated Inconel 713C. These two tests indicated a beneficial effect of sulfur in fuel which was not shown in the study conducted during the first quarter of this contract with zero or 1.0 ppm "sea salt" in air. Three tests were conducted during the current investigation using three levels of sulfur in fuel and 10.0 ppm "sea salt" in air to evaluate the effect on hot corrosion of Misco MDC-1 coated Inconel 713C.

10.3. Test Specimens

In previous studies (1) one superalloy, Inconel 713C, was evaluated in both the bare and coated condition. The coated specimens were essentially immune to attack under the conditions of exposure in our standard 5-hour test. This coating-superalloy system, Misco MDC-1 coated Inconel 713C, was selected for evaluation under

TABLE 21

COMPOSITION OF ASTM D665 SYNTHETIC "SEA WATER"

Salt (a)	Formula	Grams per liter (b)
Sodium Chloride	$NaCl$	24.54
Magnesium Chloride	$MgCl_2 \cdot 6H_2O$	11.10
Sodium Sulfate	Na_2SO_4	4.09
Calcium Chloride	$CaCl_2$	1.16
Potassium Chloride	KCl	0.69
Sodium Bicarbonate	$NaHCO_3$	0.20
Potassium Bromide	KBr	0.10
Boric Acid	H_3BO_3	0.03
Strontium Chloride	$SrCl_2 \cdot 6H_2O$	0.04
Sodium Fluoride	NaF	0.003
	TOTAL	41.953

(a) Use cp chemicals.

(b) Use distilled water.

conditions of extended test duration in the first program under this contract. A second coating-superalloy system, Misco MDC-9 coated Inconel 713C, was selected and evaluated during the second quarterly period of this contract. With the two coating-superalloy systems it was determined that an exponential relationship could be established between loss of weight of the specimens and hours of exposure, and a rate of hot corrosion calculated. All previous studies which we had conducted with uncoated Inconel 713C were with a fixed test duration of 5 hours. To complete the evaluation of the effect of sulfur in fuel on hot corrosion of these two coating-superalloy systems at the 2000 F test condition it was decided to conduct extended tests on the uncoated Inconel 713C in the same maner as with the two coatings. This study has been completed during the current quarterly period under this contract.

The Inconel 713C test specimens used in previous studies were cast from Heat No. RW063 by Misco Precision Coating Company. This supply was exhausted, and a second batch of specimens from Heat No. RW072 was obtained for programs under this contract. The chemical analysis furnished by the supplier for these two heats of Inconel 713C are shown in Table 22.

The MDC-1 and MDC-9 were applied by the supplier to test specimens of Inconel 713C from Heat No. RW-072 for these programs. These coating-superalloy systems are characterized by the following descriptions.

> MDC-1 is an aluminum coating, which was applied by a pack-diffusion process to obtain a total thickness of approximately 2 mils. It is divided about equally between an outer layer which contains non-metallic dispersions, and a diffused zone.
>
> MDC-9 is a composite coating, rich in aluminum and chromium, which was applied by a pack-diffusion process to obtain a total thickness of approximately 2 mils. The inner diffused zone is approximately 45 per cent of the total coating thickness.

The investment castings of Inconel 713C were finished by the supplier by grinding to provide specimens having a smooth uniform surface with a thickness of 0.125 ± 0.005 inches, a width of 0.500 ± 0.030 inches and a length of 2.375 ± 0.030 inches. The specimens were inspected by fluorescent penetrant (Zyglo) and X-ray to insure freedom from cracks, porosity and inclusions.

TABLE 22

COMPOSITION OF INCONEL 713C INVESTMENT CAST TEST SPECIMENS

Alloying Elements	Heat Number RW063 (a)	RW072 (b)
Nickel	Balance (71.4)	Balance (72.1)
Cobalt	<0.1	<0.1
Chromium	12.75	13.10
Molybdenum	4.49	4.51
Tungsten	...	...
Aluminum	6.40	5.73
Titanium	0.64	0.73
Manganese	<0.1	<0.1
Iron	1.43	0.93
Zirconium	0.082	0.095
Vanadium	...	...
Silicon	0.16	0.12
Boron	0.009	0.008
Sulfur	0.001	0.004
Carbon	0.15	0.12
Phosphorus	...	...
Copper	<0.1	<0.1
Cb + Ta	2.44	2.31
Cb	...	...

(a) Used in previous investigation.

(b) Used in present programs.

From physical measurements of a random sample of specimens the average surface area was calculated to be 20.27 square centimeters. From random samples of Inconel 713C specimens, and MDC-1 and MDC-9 coated Inconel 713C specimens, their average weights were determined to be:

Specimens	Average Initial Weight, mg
Inconel 713C (uncoated)	19,605
Misco MDC-1 coated Inconel 713C	19,528
Misco MDC-9 coated Inconel 713C	19,780

11. APPENDIX 4
(Test Program)

The objective of experiments under this contract has been to evaluate the effect of sulfur in fuel on hot corrosion of coatings on Inconel 713C when exposed in a marine environment. Previous tests (1) have shown that Misco MDC-1 coating on Inconel 713C was immune to attack in the standard 5-hour test over a range of temperatures and concentrations of "sea salt" in air and sulfur in fuel. An exploratory program (2) has shown that test severity could be increased to the point of coating failure by extending the duration of the tests.

The Phillips Test Facility and the Phillips 2-Inch Combustor used for these experiments are described in Section 9.1. and 9.2. of Appendix 2. The 2000 F test condition was selected from previous studies (1), and is described in Table 23, Preparation of specimens for weighing, before and after exposure, is described in Section 9.4. of Appendix 2. The composition of the fuels used for these experiments are described in Section 10.1. of Appendix 3, composition of the synthetic "sea water" in Section 10.2., and composition of test specimens in Section 10.3.

During the first two quarterly periods of this contract experiments were conducted with Misco MDC-1 coated Inconel 713C, an exploratory test was conducted with Misco MDC-7 and Misco MDC-9 coated Inconel 713C and on the basis of the exploratory tests experiments were conducted with Misco MDC-9 coated Inconel 713C. During the current quarterly period of this contract additional experiments were conducted with Misco MDC-1 coated Inconel 713C and with uncoated Inconel 713C. These programs will be discussed in detail in subsequent sections.

In all tests the three stages of the cascade (Figure 37) were loaded with six new specimens prior to the start of a test. The cyclic test was operated in periods of 5-hours with the fuel nozzle, combustor dome and liner, "sea water" injection tubes and test specimens examined at the end of each period. The basic plan for the experiments consisted of removal of one test specimen, with replacement, at each 5-hour period for the first five periods and then continuing the test to 55 hours, or until visual inspection of the specimens indicated heavy attack and the possible loss of a specimen with further exposure to the hot gases.

11.1. Misco MDC-1 Coated Inconel 713C

During the first two quarterly periods of this contract experiments were conducted with Misco MDC-1 coated Inconel 713C, using two levels of "sea salt" in air (zero and 1.0 ppm) and three levels of sulfur in fuel (< 0.004, 0.040 and 0.40 weight per cent). Two test plans were used for these experiments. The test plan, and the order of specimen removal for the first six tests, are shown in Table 24. In this program "sea water" was injected through a single tube into the quench zone of the combustor, as in previous investigations. After

TABLE 23

OPERATING CONDITIONS FOR PHILLIPS 2-INCH COMBUSTOR

Test Variables	Test Conditions
Temperature, deg. F	
Nominal Gas	2000
Exhaust Gas (a)	2039
Test Specimen, metal (b)	1970
Test Specimen, surface (c)	2074
Combustor Inlet Air	1000
Pressure, atmospheres	
Combustor Inlet Air	15
Mass Flow Rate, pounds per hour	
Air	7200
Fuel	120
Air-Fuel Ratio	60
Flow Velocity, feet per second at Test Specimen (d)	745
Test Duration, hours (e)	Varied

Notes:

(a) Values calculated using mean specific heats (9) for 100 per cent combustion efficiency. These values are confirmed by linear regression with measured temperatures in Appendix I of Reference 1.

(b) Calculated mean value from thermocouple measurements of strip temperature vs calculated gas temperature in Appendix I of Reference 1.

(c) Calculated mean value from optical pyrometer readings vs calculated gas temperature shown in Appendix I of Reference 1.

(d) Calculated value based on unblocked area in specimen holder of 1.59 square inches.

(e) Operating cycle was 55 minutes at test conditions followed by 5 minutes with fuel off.

TABLE 24

TEST PROGRAM FOR EVALUATION OF DURABILITY OF

MISCO MDC-1 COATED INCONEL 713C

Test Plan for Exposure Variables

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Order of Test
< 0.0040	0	3
< 0.0040	1.0	5
0.040	0	4
0.040	1.0	2
0.40	0	1
0.40	1.0	6

Order of Test Specimen Removal

Test Period	Test Time, hr	1	2	3	4	5	6
1	5						(a)
2	10			(a)			
3	15				(a)		
4	20		(a)				
5	25					(a)	
?	?	(b)					

Notes: (a) Remove test specimen and replace with new test specimen.

(b) Final test duration will be determined by failure of test specimen in Cascade position Number 1, as indicated by visual inspection.

reviewing the data from the six tests, the following modifications in the test rig and operating technique were made to eliminate possible sources of experimental error. To assure that "sea water" was uniformly distributed in the hot gas stream, the test rig was revised to provide for injection of "sea water" by opposed jets into the quench zone of the combustor. The "sea water" was diluted 50 per cent to maintain the same flow through the jets. To minimize any effect of position in the cascade, a system of rotation of the position of specimens was selected and a revised order of test specimen removal is shown in Table 25. At the completion of each five hour test period, the three specimen holders in the cascade were rotated, with the holder in Position 1 being moved to Position 3 and the other two holders being advanced one position. Thus specimens, with at least 15 hours of exposure, were exposed to conditions in each of the three stages of the cascade. With these modifications, one additional test was made at each of the three concentrations of sulfur in fuel, with 1.0 ppm "sea salt" in air.

A supplemental program was planned to extend the evaluation of the effect of sulfur in fuel on hot corrosion of Misco MDC-1 coated Inconel 713C to include 10.0 ppm "sea salt" in air. The test plan, and the scheduled order of specimen removal, for the supplemental program are shown in Table 26. Tests were scheduled with the three levels of sulfur in fuel used, with 1.0 ppm "sea salt" in air; however, after completing tests at the high and low levels of sulfur with little apparent difference in corrosion, it was decided to conduct a test with 4.0 weight per cent sulfur in fuel along with the 10.0 ppm "sea salt" in air to investigate the effect of increasing both the sulfur in fuel and "sea salt" in air by an order of magnitude. The length of test was reduced from the original plan because of the increased severity of corrosion with the presence of 10.0 ppm "sea salt" in air.

11.2. Misco MDC-9 Coated Inconel 713C

The test plan used for the exposure of Misco MDC-9 coated Inconel 713C specimens, to evaluate the effect of fuel sulfur on hot corrosion of this coated superalloy, is shown in Table 27. The order of test specimen removal is shown in Table 25. The test rig used in this program was modified, as discussed previously, to inject "sea water" through two opposed jets, and the specimen holders in the cascade were rotated at five hour intervals throughout the test.

TABLE 25

REVISED ORDER OF TEST SPECIMEN REMOVAL
(Misco MDC-1 Coated Inconel 713C)

Test Period	Test Time, Hours	Test Specimen Location 1	2	3	4	5	6
1	5	1_5	(2_5)	3_5	4_5	5_5	6_5
2	10	(3_{10})	4_{10}	5_{10}	6_{10}	1_{10}	2_5
3	15	5_{15}	(6_{15})	1_{15}	2_{10}	3_5	4_{15}
4	20	(1_{20})	2_{15}	3_{10}	4_{20}	5_{20}	6_5
5	25	3_{15}	(4_{25})	5_{25}	6_{10}	1_5	2_{20}
6	30	5_{30}	6_{15}	1_{10}	2_{25}	3_{20}	4_5
7	35	1_{15}	2_{30}	3_{25}	4_{10}	5_{35}	6_{20}
8	40	3_{30}	4_{15}	5_{40}	6_{25}	1_{20}	2_{35}
9	45	5_{45}	6_{30}	1_{25}	2_{40}	3_{35}	4_{20}
10	50	1_{30}	2_{45}	3_{40}	4_{25}	5_{50}	6_{35}
11	55	3_{45}	4_{30}	5_{55}	6_{40}	1_{35}	2_{50}

Notes:

( ) Remove test specimen and replace with new test specimen.
Terminate test when failure of a specimen appears imminent.

TABLE 26

SUPPLEMENTAL TEST PROGRAM FOR EVALUATION OF DURABILITY

OF MISCO MDC-1 COATED INCONEL 713C

Test Plan for Exposure Variables

Sulfur in Fuel, wt %	Sea Salt In Air ppm	Order of Test
<0.0040	10.0	1
0.040	10.0	3 (a)
0.40	10.0	2
4.0	10.0	3 (b)

(a) Test scheduled but not conducted because of small differences between test 1 and 2.

(b) Test conducted to provide a comparison with both fuel sulfur and "sea salt" in air increased by an order of magnitude over previous test program.

Scheduled Order of Test Specimen Removal

As shown in Table 25.

TABLE 27

TEST PROGRAM FOR EVALUATION OF DURABILITY

OF MISCO MDC-9 COATED INCONEL 713C

Test Plan for Exposure Variables

Sulfur in Fuel, wt %	Sea Salt In Air, ppm	Order of Test
< 0.0040	0	5
0.040	0	6
0.40	0	2
< 0.0040	1.0	4
0.040	1.0	3
0.40	1.0	1

Order of Test Specimen Removal

As shown in Table 25

11.3. Inconel 713C

The test plan used to evaluate the effect of fuel sulfur content on hot corrosion of Inconel 713C in a marine environment is shown in Table 28. The order of test specimen removal is shown in Table 25. The test rig was modified to inject "sea water" through two opposed jets, and the specimens were rotated in the cascade at five hour intervals.

TABLE 28

TEST PROGRAM FOR EVALUATION OF DURABILITY OF INCONEL 713C

Test Plan for Exposure Variables

Sulfur In Fuel, wt %	Sea Salt in Air, ppm	Order of Test
< 0.0040	0	6
0.040	0	5
0.40	0	2
< 0.0040	1.0	4
0.040	1.0	3
0.40	1.0	1

Order of Test Specimen Removal

As shown in Table 25.

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
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13. ABSTRACT An experimental investigation is in progress to determine whether the 0.4 per cent by weight of sulfur allowed in JP-5 fuel is a safe level for the protection of coated superalloys, used in aircraft-turbine engines of advanced design, when operated in a marine environment. The Phillips 2-inch combustor test facility was used to simulate environment in the turbine section of an aircraft engine with respect to temperature, velocity, pressure, and stoichiometry. Tests were conducted with a nickel-base alloy (Inconel 713C) uncoated, with an aluminum-diffusion coating (Misco MDC-1), and with an aluminum-chromium-diffusion coating (Misco MDC-9) at all combinations of three levels of sulfur in fuel (<0.0040, 0.040, and 0.40 weight per cent) with two levels of "sea salt" in air (zero and 1.0 ppm).

Exponential equations of weight-loss with time have been developed, and statistically-significant effects have been identified at a 95 per cent confidence level. In all comparisons, the removal of "sea salt" from the air significantly decreased the relative rate of corrosion; thus, indicating sea salt to be a primary causative agent of hot corrosion. The effect of sulfur in fuel varied with the superalloy coating and the absence or presence of "sea salt". In the absence of "sea salt" in air, a reduction of sulfur in fuel from the present limit to either 0.040 or <0.0040 weight per cent decreased attack with one coated superalloy and increased attack with the other. In the presence of 1.0 ppm "sea salt" in air, a reduction in sulfur to 0.040 weight per cent had no significant effect on attack; however, a reduction to <0.0040 weight per cent significantly decreased the relative rate of attack with both coated superalloys.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
CORROSION OXIDATION SULFATION TURBINE BLADES GAS TURBINE BLADES ALLOYS NICKEL ALLOYS COATINGS ALUMINUM COATINGS FUELS JET ENGINE FUELS SULFUR SEA WATER						

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